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SOUTHERN UTILIZATION RESEARCH AND

DEVELOPMENT DIVISION

of the

AGRICULTURAL RESEARCH SERVICE

UNITED STATES DEPARTMENT OF AGRICULTURE

and related work of the

STATE AGRICULTURAL EXPERIMENT STATIONS

This progress report is primarily a tool for use of scientists and administrators in program coordination, development and evaluation; and for use of advisory committees in program review and development of recommendations for future research programs.

The summaries of progress on USDA and cooperative research include some tentative results that have not been tested sufficiently to justify general release. Such findings, when adequately confirmed, will be released promptly through established channels. Because of this, the report is not intended for publication and should not be referred to in literature citations. Copies are distributed only to members of Department staff, advisory committee members and others having a special interest in the development of public agricultural research programs.

This report also includes a list of publications reporting results of USDA and cooperative research issued between July 1, 1966, and June 30, 1967. Current agricultural research findings are also published in the monthly USDA publication, Agricultural Research. This progress report was compiled in the Southern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture, New Orleans 70124, Louisiana.

UNITED STATES DEPARTMENT OF AGRICULTURE

Washington, D. C.

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INTRODUCTION

The program of the Southern Utilization Research and Development Division is an organized effort through science and technology to increase present uses and to discover and develop varied new uses for Southern farm crops. Our farmers need new markets and strengthened demand for their production. At the same time, the Nation needs the new and better products that science can create from agricultural materials. To this end the Division conducts research on cotton, cottonseed, peanuts, citrus and subtropical fruits, rice, sweet sorghum, pine gum, sweetpotatoes, cucumbers, and other vegetables.

The Division's program includes basic and applied research in the physical and biological sciences and engineering. Basic research plays a key role in uncovering new information that later may be exploited in applied research and development. When appropriate, engineers carry out pilot-plant studies of promising laboratory developments to provide engineering and cost data essential to industrial application feasibility determinations. The Southern Division has a total staff of about 597 and the in-house scientific effort in its research program amounts to approximately 199 scientific man-years. The Division consists of two Pioneering Research Laboratories (Seed Protein and Plant Fibers), eight commodity-oriented Laboratories (Cotton Finishes, Cotton Chemical Reactions, Cotton Mechanical, Cotton Physical Properties, Oilseed Crops, Food Crops, Fruit and Vegetable Products, and Naval Stores), and one Laboratory (Engineering and Development) for engineering research and development. Headquarters of the Southern Division are located at the Southern Regional Research Laboratory, New Orleans, Louisiana. The Division also has personnel and laboratory facilities at Winter Haven and Olustee, Florida; Weslaco, Texas; Raleigh, North Carolina; and Natick, Massachusetts.

Division scientists consult with specialists from other organizations during both the planning and the execution of the research, and cooperate actively with industry to facilitate commercialization and utilization of new findings. Much of the cooperation is informal, but some work is conducted under conditions described in written cooperative agreements and memorandums of understanding. Currently 60 such agreements are in effect.

The farm products with which the Southern Division deals not only provide food, clothing and industrial raw materials, but also contribute to the Nation's general prosperity and well-being. Cotton, one of the Nation's top cash crops, currently has an annual farm value of about \$1.3 billion. The retail value of cotton products is almost \$12 billion. Cottonseed, a by-product of cotton, has a farm value of \$260 million. The retail value of its products is about \$1.0 billion. Citrus grown in the U.S. has a farm value of over \$600 million; vegetables \$1.7 billion; peanuts almost \$275 million; rice about \$408 million; and pine gum about \$13.5 million. Industries processing these agricultural crops play a vital role in the Nation's economy.

There is an urgent need to maintain the traditional food, feed and industrial outlets for agricultural products and to create new and larger markets for them. Utilization research is needed to solve existing problems, to permit adjustment to important trends and to develop entirely novel operations. The opportunities are great and, as shown by past experience, the ability of utilization research to benefit the economy is tremendous. Following are a few examples of significant developments based on the research of scientists at the Southern Division.

Examples of Recent Accomplishments of the
Southern Utilization Research and Development Division

Orange Crystals for Instant Juice Used by the Armed Forces. Department scientists in cooperation with the Florida Citrus Commission have developed a new convenience food--foam-mat dried orange crystals that readily dissolve in water to yield a fresh-tasting, nutritious juice. The crystals are made from commercial frozen orange concentrate by a continuous foam-mat dryer process. Since the product is lightweight and has a shelf life of six months at 85° F., it is attractive for commercial export as well as domestic consumption. It also meets the world-wide needs of the Armed Forces, which are currently using about 1,500,000 pounds of orange crystals a year, one-third of which are produced by the new process. They are expected to use up to 5,000,000 pounds as soon as they can be supplied by industry. The potential commercial demand for the product is tremendous, not only for beverages but also for prepared dried foods or other formulations where true fruits are desired but the moisture contained in the fresh product might be a problem. Industry production is expected to increase initially to 20,000,000 pounds of orange crystals a year, equivalent to 5,000,000 boxes of oranges. At \$1.40 a box, this would be an initial annual return to the grower of \$7,000,000.

New Carbamate Agents Widely Adopted by Cotton Textile Industry for Wash-Wear and Durable-Press Finishing. New carbamates developed by Department researchers are now being used extensively for the manufacture of wash-wear and durable-press finishing agents. Five carbamates (the methyl, ethyl, hydroxyethyl, hydroxypropyl, and methoxyethyl derivatives) are currently produced commercially. Many resin manufacturers make finishing agents from carbamates supplied by four major chemical companies. A new carbamate, the isopropyl derivative, was recently announced by the Department. Consumption of carbamates in wash-wear and durable-press applications in 1965 was estimated at 4 million pounds; present annual usage is considerably higher, and the potential for further growth is excellent. The carbamates produce high-quality wrinkle-resistant finishes that are inexpensive and durable. Because the finishing agents do not yellow in high-temperature curing or upon exposure to chlorine bleach during laundering, they are equally suitable for both white and colored goods. It is estimated that carbamate finishes are used in the production of about 80% of all-white durable-press products, principally shirts, blouses, and the like.

Improved Drafting Methods Used Commercially in Cotton Processing. Basic research conducted by Department scientists has led to a better understanding of how and where fiber "hooks" are formed in mechanical processing of cotton, and has resulted in the establishing of optimum drafting directions for removing the hooks, thereby improving processing efficiency and the strength and uniformity of cotton yarn. By means of the new drafting directions, yarn breakage during spinning can be reduced as much as 20% or, alternatively, spinning rate can be increased about 10%. The increase in spinning rate, if applied to all yarns produced for printcloth alone, would reduce textile costs about \$4 million annually. Greater or lesser savings, dependent upon yarn size, would apply to other cotton products. In the case of combed yarns, the amount of cotton wasted at the comber (comber noils) can be reduced from 1 to 2%. The research findings on fiber hooks and drafting directions have recently been published, and already they are being used commercially by at least two large textile organizations to improve the quality and lower the cost of their cotton products. Other companies report that they are evaluating the findings.

High Quality Cottonseed and Peanut Flours Produced Experimentally for Human Consumption in Developing Countries. Progress has been made in cooperative research with the Agency for International Development (AID) and other organizations that are assisting developing countries to use cottonseed and peanuts in meeting their food needs. A quantity of cottonseed flour prepared at the Southern Division received long-term clinical testing in Peru as the sole source of protein given children suffering from protein deficiency and malnutrition. The physician in charge reported that this flour gave results virtually indistinguishable from those produced by skim milk products. More recently, cookies prepared by the Human Nutrition Research Division from low-fat peanut flour produced at the Southern Division were well liked by a group of 45 African women from ten different countries. Peanut flour also gave good to excellent results in such diverse preparations as beverages for babies, breads, chapatis, curry, garbanzo stew, pancakes, and noodles. Other batches of materials prepared from these oilseeds are being evaluated by various governmental, academic, or commercial agencies in the United States and in several foreign countries, including England, Canada, and Egypt. Solvent extraction systems now being investigated appear to offer promise of yielding products with acceptably bland flavor. Practical processes will be developed to enable relatively small installations in developing countries to produce high-quality flours from both cottonseed and peanuts.

New Chemical Products from Pine Gum Now Undergoing Industrial Evaluation. Three new chemical products with good industrial potential have recently been prepared from crude pine gum by Department researchers. The first is used in a new class of heat-resistant plastics. A leading manufacturer of these polymers is testing this compound as a modifier for a commercial product. Two other compounds, evaluated in the current program of screening

naval stores products as industrial chemicals, have proved to be exceptionally active as nematocides -- agents for which there is a large and growing market. Both new nematocides have attracted the attention of at least two large manufacturers of agricultural chemicals, one of which is currently evaluating one of the compounds in its own laboratories.

Examples of Recent Accomplishments of the State Agricultural Experiment Stations

Cotton Fiber to Finished Fabric in the Laboratory. Workers at the Tennessee Station in collaboration with USDA staff have developed a complete fiber to finish fabric procedure on a lab scale for studies which use only small amounts of raw stock. After constructing a miniature slasher and modifying a loom, they were able to process 10-pound lots of raw cotton stocks to test the fiber and fabric properties and study wet-finishing procedures. Identical fabrics can be woven for test purposes. These tests may be useful in marketing on the basis of fiber properties.

Continuous Pressing of Apple Juice. For centuries apple juice has been prepared by grinding the fruit and wrapping the pulp in open weave cloths to form "cheeses" which are stacked between the platens of a screw or hydraulic press. To replace this "batch process," New York food researchers have developed a continuous screw press for making apple juice. Use of the continuous press makes it possible to maintain a uniform flow of juice and requires much less labor. Principles developed will be useful in design and operation of large continuous presses.

Continuous Impregnator Developed for Fruit and Vegetable Products. Food scientists at the North Carolina Station have developed a continuous impregnator for impregnating fruits and vegetables with sugar syrup, firming agents, antioxidants and other additives. The first continuous commercial unit is now in its second season of successful operation in an apple freezing plant. The continuous unit can be placed in line easily and requires no additional operating labor. Up to 8 tons of apple slices per hour have been treated. This technique shows promise for improving the quality of canned tomatoes, sweetpotatoes and peppers.

Processing Long-Grain Rice. Texas Station researchers have investigated the effects of variety, degree of parboiling, pH, calcium lactate addition and blanching on long-grain rice canned in a liquid medium. Definite varietal effects were found. A pH of 6.5 was near optimum. Severity of parboiling was directly related to texture and appearance. Calcium lactate additions did not improve quality.

5'-Ribonucleotides Suppress Bitterness. There is great interest in the use of 5'-ribonucleotides as flavor "potentiators." Psychophysical

investigations using pair-comparisons were carried out by California food scientists to check the bitterness suppression effects of these compounds. Results indicated that ribonucleotides suppress bitterness of quinine sulfate, oxalic acid and caffeine. Ribonucleotides were as effective as the inosinate-guanylate mixture in suppressing the bitterness of aqueous quinine sulfate or oxalic acid. Inosinate-guanylate mixture was more effective than individual ribonucleotides in suppressing natural bitterness in canned spinach puree.

Freeze-Dried Sweet Peppers. Food scientists at the Puerto Rico Station have successfully freeze-dried sweet peppers (*Capsicum frutescens*). The process consists of steam treatment to inactivate enzymes, freeze-drying and packing in jars or cans. Microbial counts are reduced by steam treatments. The freeze-dried product rehydrates readily and retains good flavor. The product is especially useful in those instances in which uniformity and stability of flavor are important.

New Sweetpotato Product. A new sweetpotato product that is canned hot and congeals in the can so that it can be removed as a roll has been developed by Georgia Station food scientists. The roll can be made from sweetpotatoes that have been lye peeled, cooked, pureed and mixed with a small amount of dry milk solids. It can be sliced for serving, fried, baked and used in souffle, pies or in other ways. Advantages of processing sweetpotatoes in this form include (a) all sound field-run potatoes harvested, regardless of size and shape, can be utilized; (b) the product is uniform in color, consistency and flavor, and thereby provides uniform serving; and (c) although varieties vary in their suitability for manufacture of congealed rolls, they can be "adjusted" by blending to produce a uniform product.

Organic Acid Synthesis in Lemon Fruits. The acid content of lemon and other citrus fruits is influenced by climate, rootstock, plant nutrients and variety, and is of importance in determining fruit quality and composition. California scientists have demonstrated that organic acids can be synthesized in the fruits. Isolation of the enzyme systems from highly acid citrus fruit revealed their ability to oxidize all the tricarboxylic acid cycle intermediates and the capacity to fix carbon dioxide.

Precooked Full-Fat Peanut Flakes Developed. Short supply of protein in some areas of the world has stimulated the search for low cost methods for processing plant proteins into suitable forms for human consumption. The process, developed by South Carolina food scientists, involves converting peanuts into full-fat precooked flakes by grinding, heating with water and drum drying. The flakes, without added antioxidant, are stable against flavor changes and chemical evidence of oxidation for at least six months at 100° F. when packaged in air. They have a bland flavor and odor, and have been used as extenders in ground meat without affecting acceptability. Peanut-potato flakes having good acceptability have been prepared in several forms.

Harmful Effects of Gossypol in Cottonseed Meal Eliminated. Texas Station scientists have found that the harmful effects of gossypol in cottonseed meal can be eliminated by adding inexpensive iron (ferrous sulfate). Chemical findings indicate that the iron combines with gossypol to form a substance not absorbed from the digestive tract. The iron, added in the form of ferrous sulfate, is so inexpensive that the increase in the cost of the ration is insignificant. Feeding trials indicate that a ration of cottonseed meal and iron salts may be fed successfully to swine and broilers.

Cottonseed Flour From Glandless Seed. A white flour made from glandless cottonseed by Texas Station researchers may expand the use of cottonseed protein for human food. One of the most attractive possible uses of the flour is for production of synthetic milk. Such low-cost milk substitutes can be used to cure protein-deficiency disease in children. Characteristics of the cottonseed flour made from glandless cottonseed indicate that it may also have potential for use in the production of simulated meat products made from vegetable proteins.

AREA 1 - COTTON UTILIZATION

Problem. Cotton, the nation's most important fiber crop, is facing severe and increasing competition from synthetic fibers. Cotton is one of America's largest sources of cash farm income and still accounts for more than half of the total U. S. mill consumption of all major fibers. However, its proportionate share of the market has been slowly decreasing. The rapid growth of the synthetics at the expense of the natural fibers has been a phenomenon of the century. Expansion of market outlets for the chemical fibers has been based on vigorous research and development programs. The engineering and development programs of the chemical fiber industry are designed to capitalize on the special properties of each individual fiber as related to the functional use qualities desired in particular products; basically they involve the substitution of the newer fibers for cotton in cotton's traditional end-use markets. Expanded research to increase the utilization potential of cotton offers the most realistic opportunity for improving cotton's competitive strength as a textile fiber and for increasing cotton consumption. Basic studies of the chemical composition, physical properties and structure of cotton and modified cottons, chemical and physical investigations to improve cotton products, and research to develop the technology required for production of new and improved cotton products are basic to holding existing markets or expanding the use of cotton in new applications.

Fundamental information is badly needed in applied research to help cotton gain new and maintain old markets. Fundamental knowledge of the cotton fiber as to its structure, properties, and the mechanisms involved in chemical and physical behavior serves as a basis and a guide in the design and improvement of processing machinery, mechanical and chemical processes, and in the development of new and improved cotton yarns, fabrics, finishes, and treatments. Many chemical and physical treatments, as well as textile organizations and machine designs, offer a basis for the improvement of cotton quality or lowering of processing costs. Exploratory chemical and physical research is needed to determine the true potential of such approaches prior to undertaking extensive developmental research or the construction of prototype machinery. New or improved mechanical processing methods and textile machinery are urgently needed by the cotton industry. A major problem at the present time is the utilization of the large quantities of low and high Micronaire cottons that are now accumulating in public and private stocks. Cotton products with various special properties and processes for their production must likewise be developed to meet the serious competition of synthetic fibers and other competitive materials in numerous end uses. Some of the more important types of products that need to be developed or improved include: wash-wear textiles; wash-wear, durably pressed garments with adequate abrasion resistance; stretch and bulked products; weather- and rot-resistant fabrics; flame-resistant products; soil-resistant textiles; fabrics with water and/or oil repellency; textiles with

improved luster; textiles with multifunctional properties; and insect-resistant bags for storage and shipment of food commodities. An essential part of such a utilization research program is the development of new and improved methods and instruments for measuring the physical and chemical properties of cotton and its products.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving organic chemists, analytical chemists, physical chemists, physicists, microscopists, chemical engineers, mechanical engineers, statisticians, cotton technologists, textile technologists, and textile engineers engaged in both basic and applied research to help cotton gain new and maintain old markets. Cooperation is maintained with greige goods mills, textile mills, finishers, chemical manufacturers, manufacturers of textile machinery, cotton merchants, textile research institutes, and industry associations in connection with the research. The Crops Research Division, ARS, and the Cotton Division, C&MS, also cooperate, particularly in the procurement of cotton with special fiber properties and of known history. Cooperation is also maintained with the Market Quality Research Division, ARS, to insure coordination of effort in any related research.

Basic research on the chemical composition, physical properties and structure of cotton and its products is carried out at the Southern Regional Research Laboratory, New Orleans, Louisiana. This research provides information on the relationship of properties and structure to behavior of cotton in mechanical and chemical treatments, essential to an understanding of the performance of fibers during processing and in textile products. The development of new methods and instrumentation for measuring physical and chemical properties of cotton and its products is an integral part of the program. Also included is the research of the Plant Fibers Pioneering Research Laboratory to obtain basic information on the supermolecular structure of plant textile fibers; and to relate information of polymer and fiber structure to the mechanical and textile behavior of fibers. Additional basic research on chemical and physical properties and structure of cotton is being carried out: (1) under contracts at Stanford Research Institute, South Pasadena, California, on development of a method for counting neps in cotton at various stages of textile processing; and on development of a research instrument for accurately and automatically determining length, length distribution and diameter of cotton fibers; at the University of Tennessee, Knoxville, Tennessee, on investigations to determine the effects of fiber extensibility on fiber breakage in mechanical processing; and at the Polytechnic Institute of Brooklyn, Brooklyn, N. Y., on relationship of molecular size, nature, shape, conformation, and configuration of organic nonaqueous compounds to their swelling power on cotton cellulose; and (2) under a grant at Georgia Tech Research Institute, Atlanta, Georgia, on elucidation of the role of fiber morphology on frictional behavior.

Chemical and physical investigations to improve products is also conducted at New Orleans, Louisiana. Exploratory chemical and physical research is carried out as a basis for improving mechanical and chemical processing, and developing new and improved yarns, fabrics, finishes, and treatments. One phase of the research--exploratory investigation of reversible chemical reactions to obtain information basic to the development of a commercially feasible reversible crosslink--is conducted with cooperation and support by the Cotton Producers Institute. The International Lead Zinc Research Organization cooperates in and supports exploratory research to impart useful properties to cotton through application of selected lead and other metal compounds. Additional exploratory chemical and physical investigations are also being carried out: (1) under contracts at Harris Research Laboratories, Inc., Washington, D. C., on the development of finishes for cotton fabrics to render them more rapid drying; at Southern Research Institute, Birmingham, Alabama, on the development of wash-wear cotton fabric with improved moisture absorptivity by use of reactive swelling agents; at Bjorksten Research Laboratories, Inc., Madison, Wisconsin, on the effect of resin thermoplasticity or thermosettability on the resistance of treated cotton fabrics to abrasion; and at New York University, Bronx, New York, on polymer encapsulation of cotton fibers as a means of providing new and useful products; and (2) under grants at Textile Research Institute, Princeton, New Jersey, on crosslinking of chemically modified cotton to obtain cotton fabrics with an optimum combination of resilience and thermoplasticity; and at the University of Arizona, Tucson, Arizona, on correlation of surface microtopography of treated and untreated cotton fibers with resistance to soiling of cotton textiles.

Research on process and product development is carried out at New Orleans, Louisiana. Major facets of the work include the development of: improved procedures for mechanical processing of cotton, new and improved textile machinery (opening through carding), and cotton products with special properties for various end uses. The principal types of products currently involved in the latter research include: wash-wear yard goods and garments, including those with durable-press features; weather- and rot-resistant fabrics; soil-resistant textiles, including those with water and/or oil repellency; flame-resistant products; textiles with multifunctional properties; stretch and bulked products; and insect-resistant bags. Pilot-plant evaluations of promising laboratory processes and products are carried out, and cost estimates are made to aid industrial establishment of various research developments. Close cooperation is maintained with cotton textile machine manufacturers and cotton textile processors in the evaluation of experimental textile machinery developed in the research, and in the establishment and dissemination of engineering specifications for the commercialization of the new machinery. The research to develop cotton fabrics with improved resistance to outdoor weathering is cooperative with the Foundation for Cotton Research and Education (affiliated with the National Cotton Council of America) and the Canvas Products Association International. The Cotton Producers Institute cooperates in and supports research to develop optimal cotton fabric structures for men's trousers and

dress suits. Research on cotton batting is conducted in cooperation with the National Cotton Batting Institute, Textile Waste Association, National Cottonseed Products Association and the Foundation for Cotton Research and Education (affiliated with the National Cotton Council of America). The Stored-Product Insects Research and Development Laboratory, Market Quality Research Division, ARS; bag manufacturers; and the Textile Bag Manufacturers Association cooperate in the work to develop improved insect-resistant cotton bags.

Additional research on process and product development is being carried out under contracts at Auburn Research Foundation, Inc., Auburn, Alabama, to determine optimum processing procedures for cotton differing in tensile and elastic properties and relate these properties to mechanical processing performance and yarn and fabric properties; and to develop mechanical-chemical surface treatments for fabrics to improve abrasion resistance of durably pressed cotton garments; at Southern Research Institute, Birmingham, Alabama, on investigation of interfacial and graft polymerization procedures for producing weather-resistant cotton textiles with improved physical properties; and at Fabric Research Laboratories, Inc., Dedham, Massachusetts, on development of improved coated cotton fabrics with optimum strength-weight characteristics for outdoor uses; and on development of methods for improving the dimensional stability of abrasion-resistant durable-press fabrics made from blends of resin-treated and untreated cotton fibers.

Other research on chemical and physical properties and structure of cotton is in progress under grants of P. L. 480 funds to the following foreign institutions: Fiber Research Institute, T.N.O., Delft, Holland, for an investigation of the fundamental mechanisms and bonding forces that could be used to improve tensile strength and other physical properties of cotton textiles (project duration - 5 yrs.); Ahmedabad Textile Industry's Research Association, Navrangpura, Ahmedabad, India, for the study of the relation between the fine structure and mechanical properties of cotton fibers by swelling and stretching treatments (project duration - 5 yrs.); and for a study of the physical chemistry and thermodynamics of solution and vapor phase adsorption on and in the cotton fiber (project duration - 5 yrs.); University College, University of Wales, Cardiff, Wales, for an investigation of the nature and mechanism of the chemical effects of ultraviolet light on cotton cellulose and related compounds (project duration - 3 yrs.); Indian Central Cotton Committee, Bombay, India, for investigation of the microbial decomposition of cellulose with special reference to the effect of Indian bacterial organisms on cotton and cotton fabrics (project duration - 4 yrs.); Juan de la Cierva School of Technical Investigations, Barcelona, Spain, for a study of the measurement of "total hairiness" of cotton yarn and the determination of mechanical factors contributing toward its formation (project duration - 5 yrs.); The Cotton Silk and Man-Made Fibres Research Association, Shirley Institute, Didsbury, Manchester, England, for a study of the effect of swelling agents on the fine structure of cotton (project duration - 5 yrs.), and for an investigation of chemical modifications of cotton fabrics involving control of lateral molecular order and

distribution of crosslinks (project duration - 3 yrs.); Shri Ram Institute for Industrial Research, Delhi, India, for a fundamental investigation of moisture sorption and desorption by variously crosslinked cotton celluloses over the entire humidity range (project duration - 5 yrs.); State University of Ghent, Ghent, Belgium, for a fundamental study of the nature and origin of reversals in cotton fibers and their relation to mechanical properties of the fibers (project duration - 4 yrs.); Swiss Federal Institute of Technology, Zurich, Switzerland, for a study of the chemistry and structural nature of the bonds formed between formaldehyde and cellulose in formaldehyde-treated cottons (project duration - 5 yrs.); Lodz Polytechnic College, Lodz, Poland, for an investigation of the mathematical and theoretical aspects of the relationship between the fiber length distribution of cotton specimens before and after sample preparation (project duration - 3 yrs.); Ministry of Commerce and Industry of the State of Israel, Jerusalem, Israel, for a fundamental investigation of the geometry of wrinkles as they affect the rating of acceptability of ease-of-care treated cotton fabrics (project duration - 4 yrs.); and Central Laboratory, T.N.O., Delft, Holland, for a study of the influence of yarn geometry on the response of the structural elements of chemically treated cotton fibers to stress and deformation (project duration - 3 yrs.).

Chemical and physical investigations to improve products are also in progress under grants of P. L. 480 funds to the following foreign institutions: The Hebrew University of Jerusalem, Jerusalem, Israel, for the synthesis and determination of properties of new aziridinyl phosphorus compounds having potential for use in the treatment of cotton (project duration - 3 yrs.); Indian Central Cotton Committee, Bombay, India, for an investigation of the preparation of radioresistant and radiosensitive celluloses (project duration - 5 yrs.); Ministry of Commerce and Industry of State of Israel, Jerusalem, Israel, for a fundamental study of the oxidation of cotton and crosslinked cotton by various oxidizing agents (project duration - 3 yrs.); Chalmers University of Technology, Gothenburg, Sweden, for a basic investigation of the behavior of cotton subjected to aerodynamic forces (project duration - 3 yrs.); Shri Ram Institute for Industrial Research, Delhi, India, for a fundamental investigation of heat and mass transfer rates in the drying and curing of resin-treated cotton textiles by countercurrent solid-gas contact systems (project duration - 5 yrs.), and for investigations of the correlation between several important physical properties of woven cotton apparel fabrics and their performance in actual service tests, to obtain information needed for the improvement of cotton textiles (project duration - 5 yrs.); Ahmedabad Textile Industry's Research Association, Navrangpura, Ahmedabad, India, for investigation of means to minimize fiber hooked ends in cotton card and drawing slivers (project duration - 4 yrs.), for an investigation of factors affecting drafting in the direct sliver spinning system (project duration - 5 yrs.), and for the development of a stochastic model for determining the efficiency of drafting independent of the size of fibrous strands (project duration - 5 yrs.); Juan de la Cierva School of Technical Investigations, Barcelona, Spain, for an investigation of the effect of fiber properties on drafting tenacity during spinning of cotton

and the interrelationships between fiber properties, drafting tenacity, yarn properties, and end breakage, to obtain basic information related to processing properties in the utilization of cotton (project duration - 4 yrs.); South India Textile Research Association, Coimbatore, India, for an investigation of the effects of atmospheric conditions during the spinning of cotton yarns on yarn properties and spinning efficiency (project duration - 5 yrs.); Fiber Research Institute, T.N.O., Delft, Holland, for fundamental investigations to obtain information needed to predict the performance of cotton yarns during weaving (project duration - 3 yrs.); Swedish Institute for Textile Research, Gothenburg, Sweden, for investigation of the mechanism of crease formation and recovery in ease-of-care treated cotton fabrics (project duration - 4 yrs.); and Bombay Textile Research Association, Bombay, India, for a study of factors affecting curling and bursting of preponderantly warp- and filling-faced cotton fabric structure during processing of cotton into end-use products (project duration - 5 yrs.).

The Federal in-house scientific effort devoted to research in this area totals 105.1 scientific man-years. Of this number, 23.7 is devoted to chemical composition, physical properties and structure, 23.7 to chemical and physical investigations to improve products, and 57.7 to technology--process and product development. The domestic contract and grant research involves an additional 14.2 man-years, 5.0 being on chemical composition, physical properties and structure, 4.9 on chemical and physical investigations to improve products, and 4.3 on technology--process and product development. P. L. 480 research involves 28 grants, of which 14 are on chemical composition, physical properties and structure and 14 on chemical and physical investigations to improve products.

The following lines of work were terminated during the year: (1) Investigation of the fluorescence spectra of native and modified cottons; (2) Investigation of new solvents for molecular weight determination of cellulose, to obtain basic information needed to improve cotton products and thereby enhance the utilization of cotton (P. L. 480 project); (3) Investigation of the configurational interactions between fibers and yarns in the region of local deformations in woven cotton cloth; (4) Investigation of factors influencing comfort in cotton apparel fabrics; (5) A fundamental investigation of setting reactions for cotton fabrics and garments, to develop information basic to the improvement of cotton products, thereby increasing the utilization of cotton (P. L. 480 project); (6) A study of the effect of the soiling environment on the soiling tendency of a series of cotton finishes; (7) A fundamental study of the preparation and properties of phosphazene and phosphoryl chloride derivatives having potential for reaction with cotton cellulose, to obtain information needed in the development of new useful products from cotton, thus increasing its utilization (P. L. 480 project); (8) Evaluation of stretch-type cotton yarns (prepared by back-twisting and falsetwisting techniques) in knitwear; (9) Determination of optimum yarn constructions, knitting structures and prefabrication design for producing stretchable articles of knitted cotton wearing apparel by slack mercerization; (10) Investigation of the physics of seam pucker in

relation to fabric structure to develop improved sewing thread for wash-wear cotton products; and (11) Determination of the effect of high production carding on fiber length distribution and formation of fiber hooks in cotton. The first five lines of work were under Chemical Composition, Physical Properties and Structure, the next two under Chemical and Physical Investigations to Improve Products, and the rest under Technology--Process and Product Development.

PROGRAM OF STATE EXPERIMENT STATIONS

A total of 3.8 scientific man-years is devoted to this area of research.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition, Physical Properties and Structure

1. Fundamental Investigations of Adsorption and Swelling Phenomena in Native and Modified Cottons. Differences in internal structure of cotton fibers from fabrics crosslinked by different techniques have been indicated by measurements of surface areas and pore-radii distributions by gas adsorption methods. The data appear to parallel findings of changes in tensile properties, as well as observations made by electron microscopical techniques. The surface areas of cottons modified to produce crease resistant textiles are significantly smaller than those of unmodified cottons. Of various cotton samples modified to improve crease recovery, only those treated with formaldehyde by the Form W method showed less than fifty percent decrease in surface area after treatment. Rowden, Deltapine, and Pima S-2 cottons in the yarn form, when studied by gas adsorption techniques after dewaxing only or after dewaxing then scouring, showed greater differences in surface area and pore characteristics between treatments than between varieties. Information from this research should be useful in predicting properties of treated cottons and, thus, in selecting the most efficient agents and processes for obtaining desired end-use properties. (S2 1-209(Rev.)).

A basic investigation of the influence of internal and external restraint on the capacity of cotton fibers to sorb swelling solutions is in progress. Recent work has shown that both the method and the degree of crosslinking of cotton fibers with formaldehyde influence the extent to which the treated fibers sorb sodium hydroxide solution. The alkali sorption values of yarn segments of cotton fabrics crosslinked by the Form W and W' processes to levels below 0.8% formaldehyde were found to be greater than that of the uncrosslinked control, reaching a maximum at about 0.3% formaldehyde. For samples crosslinked in systems employing strong solutions of acetic acid or phosphoric acid, the sorption values decreased as the formaldehyde content increased, with no indication of a rise in sorption. The alkali treatment (15% sodium hydroxide) in the sorption tests raised the hygroscopicity of all crosslinked specimens of less than 1% formaldehyde to a considerably

higher level than their original moisture regain values and to a level even greater than that of the uncrosslinked control. (S2 1-249; S2 1-301).

In contract research at the Polytechnic Institute of Brooklyn, changes in fine structures of cotton and rayon celluloses caused by immersions in various organic liquids have been determined by means of X-ray diffractograms. From small angle X-ray diffractograms of the celluloses in the liquids it was found that ethylene glycol at room temperature and dimethylformamide at 80°C cause as much swelling in cotton as a 5% aqueous sodium hydroxide solution at room temperature. Dimethylformamide at 25°C causes some swelling in cotton but not in rayon. Measurements of the uptake and loss of dimethylformamide at various temperatures will be used to calculate energies required to make or break hydrogen bonds formed between cotton and the liquid. Data on swelling of cotton as revealed by several methods are currently being correlated. The findings should contribute to a better understanding of mechanisms involved in the swelling of cotton. (S2 1-225(C)).

Good progress has been made in a study of the physical chemistry and thermodynamics of solution and vapor phase adsorption on and in the cotton fiber under a P. L. 480 project at the Ahmedabad Textile Industry's Research Association, Ahmedabad, India. A proposed theoretical model for the equilibrium of cellulose fiber-aqueous system has been extensively tested through a number of adsorption isotherms for dyeing cotton with selected dyes. Thermodynamic quantities such as affinities, heats and entropies of dyeing have been evaluated on the basis of the theoretical model. Vapor phase adsorption studies using nitrogen as the adsorbing molecule have indicated that there is a very marked increase in the surface area when fibers are swollen with water. The work has been extended to detect differences in adsorption of formaldehyde in Form W and Form D processes, and on specific surface areas of chemically modified cottons. It has been shown that the Form W process does not interfere with the nature and extent of fiber surface available for dye adsorption, while Form D does. This suggests that Form W causes open network stabilization of the amorphous regions of cotton whereas Form D preserves the network in a more collapsed unswollen state. Basic information being obtained is expected to be of value in improving both wet and vapor phase treatments of cotton fabrics for easy-care applications. (UR-A7-(20)-46).

A study of the moisture sorption and desorption by crosslinked cotton over the entire humidity range as related to the state of swelling under which the cellulose is crosslinked is getting well underway in a P. L. 480 project at the Shri Ram Institute for Industrial Research, Delhi, India. Standard cellulose prepared from an Indian cotton has been crosslinked with formaldehyde at different degrees of swelling using various acidic catalysts. Moisture sorptions determined over a range of relative humidities have revealed maxima in the range of 0.35 to 0.50% formaldehyde at all levels of relative humidities studied for swelling levels of 2.5% and 16%. At 7% swelling level, cellulose degradation was observed. Calculations of physical

properties such as heats of sorption, pore size distribution and internal surface are being made to elucidate the mechanism of sorption phenomena. The studies will be extended to other levels of swelling and formaldehyde content. The basic information being obtained will be of value in treatments of cottons involving formaldehyde crosslinking reagents for the finishing of fabrics. (UR-A7-(20)-59).

2. Basic Studies of the Relationships of the Structural Arrangements Within Cotton Fibers to the Physical Properties of Native and Modified Cottons.

Microscopical evaluations of various types of modified cottons have continued. Electron micrographs of ultrathin sections of irradiated cottons grafted with various methacrylate polymers indicated that the polymers had entered into the ultrastructure of the cellulose and formed spaces between the layers. Other experiments showed that when ultrathin sections of partially acetylated cotton, 26% acetyl, were deacetylated, the fibrillar structure reverted to that of untreated cotton. Electron microscope observations of cotton samples treated with a vapor phase carbamate finish and then exposed to butanol hemiformal indicated that the cottons had crosslinks at the level of section thickness (about 500-600 Å). Other work showed that acetylated cottons of low degrees of substitution prepared by vapor phase techniques did not swell or form layers in methacrylate embedding medium as do many cellulose esters. It was also found that in comparison with a control, the average refractive indices of aliphatic cellulose esters were lower, whereas those of the aromatic esters were higher. (S2 1-263; S2 1-302).

Research has been initiated to investigate the molecular orientation of native and modified cotton fibers and its relationship to the physical properties of modified cottons. Results obtained from polarized light studies of ten different genetic varieties of cotton showed that the spiral angle of unconvoluted cotton fibers was a constant regardless of genetic variety. X-ray angles of fiber bundles swollen in caustic approach this constant value, indicating that the X-ray angle of untreated cottons involves the true spiral angle as well as an angle of fiber convolution. (S2 1-307).

In further research on plastic and oriented states of cotton cellulose, the shape imparted to cotton fabric and yarn after swelling and plasticizing with aqueous benzyltrimethylammonium hydroxide has been greatly stabilized by subsequent application of certain resins which form three-dimensional polymers. Aftercuring of the creased areas of plasticized fabric with trimethylolmelamine in the absence of crosslinking catalyst overcame both the puckering that occurred at boundaries of treated areas and the stiffness imparted. No strength losses appear to have resulted from the treatment. The coiling imparted to plasticized yarn was very greatly increased by similar treatment with either trimethylolmelamine or tris(aziridinyl)phosphine oxide. Swelling of parallelized cotton fiber bundles by wetting with 35% benzyltrimethylammonium hydroxide has been found to convert the cotton cellulose to a form that is completely nondiffracting towards X-rays and therefore amorphous--yet the cotton retains its fibrous form. Removal of the quaternary hydroxide by washing causes recrystallization of cellulose even while the

cotton is wet. However, cotton treated with mixtures of quaternary hydroxide and sodium hydroxide is apparently converted to a crystalline form different from cellulose I or II. (S2 1-284).

Studies of cotton fibers grown in controlled environments (see project S2 1-217(C) below) have shown that the diurnal ring structure and structural reversals of the fibers apparently have no large influences on tensile and mechanical properties. Perhaps the arrangements and movements of fibrils in the cell walls play a greater role than heretofore believed. No evidence was obtained to conclude that there is a relationship between ring structure and chain length of the cellulose. The reversals were found to be related to the genetic origin of a cotton as well as environmental factors. Fibers grown with water deficiency had a lower reversal frequency than those grown under normal conditions. The location where cotton fibers break is controlled by structural features in addition to the reversal, even though the reversal is the preferred position of fracture. Low temperature of growth tended to increase fiber length, elongation, X-ray angle, and moisture regain and to decrease maturity, strength, and crystallinity. In research on field grown cottons, it was found that reversal frequency showed some correlation with single fiber strength and elongation. Mercerization and resin treatment were about equally effective in decreasing the proportion of fibers breaking at the reversals. When resin treatment followed mercerization, the effects were essentially cumulative. (S2 1-208(Rev.)).

Completed work by the contractor (Texas Agricultural Experiment Station) in which cottons were grown in chambers under controlled conditions demonstrated that environment, temperature, light and moisture affect both plant response and fiber structure. Boll growth period is inversely related to temperature. However, despite the longer growth periods, low temperatures produce fibers with thinner walls. The plants respond to change in rhythmic cyclic conditions by temporarily wilting. At high temperatures, flowering is not affected but flowers are sterile and boll retention is very low. (S2 1-217(C)).

A fundamental study of the nature and origin of reversals in cotton fibers and their relationship to mechanical properties of these fibers is continuing in a P. L. 480 project at the University of Ghent, in Belgium. Previous examination of a number of U. S. cottons of several varieties grown in different locations indicated that a definite, though small, effect on distances between reversals is due to growth location, while a much larger effect is due to variety. Also certain botanical archetype cottons exhibited no reversals at all, an important genetic observation. Measurements of mechanical properties of the fibers made with a single fiber tensile tester have definitely shown that fiber reversals represent points of weakness. The strength at reversals ranges from about 70 to 98% of the strength between reversals. Examination of U. S. cottons of three varieties grown at 12 locations has led to the conclusion that variety is the most significant factor in determining fiber reversal density. Fundamental information of the type being obtained in this project will be useful in relating the

structure of cotton fibers to their usefulness in cotton products and will assist plant breeders in developing better cottons through the use of genetic information being obtained. (UR-E4-(20)-1).

Research has continued under a P. L. 480 project at the Fiber Research Institute, T.N.O., in Holland, to investigate the fundamental mechanisms and bonding forces that could be used to improve the tensile strength and other physical properties of cotton textiles. Work is progressing to determine the physical and mechanical properties of combed yarns after treatment with various media that strongly swell cotton fibers. Progress in this area has necessitated the development of satisfactory methods for using dry organic liquids for fluid exchange procedures in the cotton fibers. Methods for controlling humidity while conducting fiber measurements, including apparatus to permit measurements in an absolutely dry atmosphere, have been developed. Information growing out of research under this project is expected to be useful in improving processing treatments to yield cotton fabrics having improved strength characteristics. (UR-E19-(20)-12).

3. Elucidation of Mechanisms of Physical Damage to Cotton Due to Mechanical, Chemical, Physical or Biological Actions. In contract research at the University of Tennessee to determine the effects of fiber extensibility on fiber breakage in mechanical processing, it has been found that the interaction of fiber properties when assemblies of cotton fibers are placed in torsion masks the torsional differences of the cottons. Torsional measurements will have to be made on single fibers rather than by the bundle method. The coefficient of friction of cotton fibers has been found to depend not only on the surface of the fiber but also on that of the other contacted material, such as other cotton fibers, glass and steel. The magnitude and ranking of the values changed with the particular surfaces involved. It was established that the decreased strength of yarns made from cotton fibers exposed to various heat and chemical treatments was caused principally by increase in yarn nonuniformity rather than by decrease in fiber strength. (S2 1-221(C)).

Work is in progress at the Technological Laboratory of the Indian Central Cotton Committee, Bombay, India, under a P. L. 480 grant to investigate the microbial destruction of cotton fibers and fabrics that occurs in exposure during outdoor uses. A number of bacterial and fungal cultures have been isolated from cotton fabrics exposed to outdoor environments in the Bombay area. Taxonomic studies have been made on these isolates and they have been screened for cellulolytic activity. Active species have been grown on carboxymethyl cellulose and on powdered cellulose to assay their production of cellulase enzymes. This work is being extended to an assay of enzymes produced when the organisms are grown on dewaxed, undegraded cotton substrates and to growth in media containing sea water. A study eventually will be made of the mechanisms by which these organisms cause rotting or tendering of cotton fabrics. Basic knowledge of this type will be useful in devising improved treatments to prevent or minimize biological degradation of cotton fabrics. (UR-A7-(20)-32).

4. Investigation of the Structural and Compositional Changes Occurring During Chemical and Physical Modification of Cotton Cellulose. Pioneering research on plant fibers has been continued along a number of lines by the Plant Fibers Pioneering Research Laboratory. The objective of this Laboratory is to obtain basic information on the fine or supermolecular structure of plant fibers; and to relate the information of polymer and fiber structure to the mechanical and textile behavior of fibers.

Studies of the effect of environmental conditions during growth on the fine structure of cotton fibers are being continued. Evaluation tests have been completed on several series of cottons grown in environmental chambers at Texas A&M University (contract project S2 1-217(C)). Bolls grown under continuous illumination at a constant temperature of 90°F matured very rapidly; they opened 30-35 days after blossom--much sooner than usual--and the cellulose content of the fibers increased more quickly with age of boll. Accessibility as measured by enzyme attack and dye sorption decreased rapidly. By contrast, bolls grown at a constant temperature of 80°F matured more slowly. The fibers were rather thin-walled up to the date of boll opening.

Molecular weight distribution curves were obtained by gel permeation chromatography of selected nitrated samples. It was found that the temperature during growth of the cottons affected the average degree of polymerization (DP) of the cellulose in the fibers. Continuous high temperature (90°F) produced more high-DP material and less of lower DP than did a continuous low temperature (70°F). Alternating temperatures tended to produce lower DP cellulose; thus, an 80°-70°F alternation of temperature resulted in nearly as much very low DP fraction and more of intermediate DP fraction than did constant 70°F, and a 90°-60°F alternation shifted the peak of the DP distribution curve downward.

Fresh and preserved samples of fibers were observed with the electron microscope in cooperative experiments. It was shown that freeze-dried fibers had nearly the same appearance in cross-section as fresh (still moist) fibers, but that preservation in methanol caused severe shrinkage.

Characterization of the supramolecular arrangement of cellulose and its physical and chemical modifications by X-ray diffraction techniques has continued to yield basic information on the nature of the response of the fiber to technologically important processes. Measurements of the extent and type of crystalline cellulose, the dimensions of the crystalline areas, and the geometry of the crystal unit cell after chemical modification are of particular interest.

It has been possible, by the application of a computer-programmed multiple regression technique, to estimate the fractions of crystalline cellulose I and II and amorphous cellulose in materials of mixed crystalline lattice types. This analysis provides information on the extent of conversion from the cellulose I to the cellulose II lattice in mercerization procedures as

well as an indication of the overall change in the amount of crystalline cellulose present. The reliability of the estimates is approximately 5%, which is satisfactory for many technological purposes. The technique could be extended to include cellulose III and IV estimations if desired.

The crystallite dimensions of cotton cellulose, whether in the cellulose I, II, III, or IV crystalline modification, appear to change to a relatively small extent under conditions of strong acid hydrolysis. The average crystallite lengths, measured for the first time after drastic hydrolysis, differed by less than 20% from that of the original native cotton (approximately 200 Angstrom units) from which the crystalline modifications and their hydrocelluloses were derived. Crystallite width appears to be slightly increased by acid hydrolysis. Thus, the regions of high lateral order in cotton exhibit remarkable stability under attack by acid.

The effect of introducing various side groups into cellulose on its crystalline arrangement was studied. In the esterification of cotton with propionyl chloride in pyridine, a significant amount of the propionyl-propionate ester moiety was produced, and the product showed poor crystallinity; this was not improved by heat treatment. If the side group introduced was sufficiently bulky, crystallization was again prevented from taking place to any great extent. In nearly all of the derivatives examined, the increases in spacing between cellulose chains were greatest in the (101) interplanar dimension; that is, the chains were moved apart to the greatest extent in the direction corresponding to the planes of the anhydroglucose rings. In some cases, the (101) spacing was increased from its original value of 7.5 Angstrom units to as much as 20-25 Angstrom units. That such enormous lattice distortions are achieved without loss of fiber integrity indicates that a wide variety of large chemical groupings may be employed in cellulose to give textile products with useful properties.

In continuation of a study to characterize and more fully interpret the microfibrillar organization of cellulose fibers, gel permeation chromatography of the nitrated cellulose has been employed to obtain molecular weight distributions and related parameters. Much of the effort thus far has been directed to methodology and calibration. Prepared samples of native cellulose from yarn and fabric showed a fairly broad distribution of degree of polymerization (DP), peaking at 21,000, with polymolecularity ratios (PMR) of 1.6 - 1.9. The DP distributions of the "leveling off" hydrocelluloses prepared from these materials were greatly broadened, with a PMR of 3.7 - 4.0 and peaking at 560-600. The curves were fairly symmetrical; they were unimodal for the initial celluloses but for the hydrocelluloses there was an indication of a second peak at a rather low DP. An acetylation grade cotton linters, although peaking at DP 10,000, showed a DP distribution only slightly broader (PMR 2.7) than that of cotton fiber, but with a long "tail" in the low DP region reaching down to 100.

In other work, the Chatterjee method for reaction kinetics of pyrolysis by thermogravimetry was applied to two control cellulose samples and to five of

their triphenylmethyl ether derivatives which varied in degree of substitution from 0.31 to 0.79 per anhydroglucose unit. Three different methods of treating the data from the thermograms indicated zero or low order reaction at the beginning of pyrolysis (ca. 240°C), followed by first or higher order beginning at about 270°C. The tar phase of the reaction was completed at about 350°C. Usually, only one extended region of first order decomposition was detected in the control celluloses but two or more, with different Arrhenius parameters, were detected in the ether derivatives. One of the first order regions, usually the intermediate one with very low activation energy, was associated with scission of the trityl group (285-310°C). In certain cases the char region (350-500°C) was examined by the same techniques but without discovery of a recognizable kinetic law. The results are in substantial agreement with the chain mechanism of cellulose pyrolysis proposed by Chatterjee and Conrad.

Further studies of the modification of cellulose structure by optically active substituents have been made. The objective of this research is to determine to what extent a change in the configuration of a reactant can affect the rate of formation of a cellulose derivative and the physical (textile) properties of that derivative. The optically active acid chlorides derived from menthoxyacetic, menthyl hydrogen terephthalate, and β -methyl hydrogen camphorate were reacted with cotton yarns in pyridine at 100°C to obtain the cellulose esters. All of the acid chlorides were very reactive and weight gains of 100% or more were readily achieved in less than two hours. None of the esters showed any tendency to dissolve in its reaction medium. No differences in reactivity toward cellulose could be detected between the enantiomorphic forms of menthoxyacetyl chloride, or between the enantiomorphs of menthyl terephthaloyl chloride. Results with the β -methyl camphoryl chlorides were difficult to interpret primarily because of the ease with which they were isomerized to give a mixture of β -methyl camphoryl chloride and β -methyl isocamphoryl chloride.

Side products in the reactions of cotton cellulose with propionyl chloride and with phenylpropionyl chloride have been investigated. In the reaction of cotton cellulose fiber with propionyl chloride in pyridine-dimethylformamide medium, the degree of substitution based on weight gain was much higher than that based on saponification. 3-Pentanone, a compound arising from the pyrolysis of α -propionylpropionic acid, was identified among the pyrolysis products of the propionylated celluloses. Quantitative gas liquid chromatography showed that 25% of the hydroxyl groups of the cellulose were substituted with an α -propionylpropionyl moiety, the remaining 75% with propionyl groups only. In the heterogeneous esterification of cotton cellulose with β -phenylpropionyl chloride in a pyridine-dimethylformamide medium, the product was a mixed ester, the α -hydrogen atom of the primary ester in some of the chains being substituted by another phenylpropionyl group.

Research on the preparation and properties of cellulose furoate has indicated that strong, resilient cellulose furoate yarns can be produced. Cotton yarns

reacted with furoyl chloride in a mixture of dimethylformamide and pyridine to degrees of substitution of 0.35, 0.70, 1.00, 1.71, and 2.01 were tested. The breaking strength of the yarns, based on the tex of the control, remained at 80% or more. The elongation increased with degree of substitution and reached about 260% of that of the control. When the cellulose furoate was examined by X-ray diffraction it was found that the original cellulose crystal is very much distorted by the treatment: $\beta = 48^\circ$; dimensions along the a, b, and c axes were 18.1, 10.3, and 13.6 Angstroms, respectively. The corresponding characteristics of the unreacted cellulose were: $\beta = 62^\circ$, a, b, and c were 8.14, 10.3 and 9.14. (SU P 2).

In addition to the aforementioned research conducted in the PF Pioneering Research Laboratory, several related lines of in-house research were conducted elsewhere. In one of these--a spectroscopic study of molecular structural changes produced by the chemical modification of cotton--the far infrared spectra (in the region $600\text{--}300\text{ cm}^{-1}$) of additional compounds structurally related to cellulose were obtained and tentative band assignments made. These band assignments may prove useful in assigning bands in the spectrum of cellulose in this region. Techniques developed for obtaining spectra of pyrolysates of chemically modified cottons appear promising for studying structural changes. However, further experiments will be necessary to determine the usefulness and reproducibility of the observed spectral changes. The most satisfactory technique found for obtaining spectra of the acid hydrolyzates of modified cottons involves the use of polyethylene discs upon which the hydrolysis product is evaporated. In studies of several series of weathered cotton fabrics, there was no apparent correlation between time of weathering and crystallinity index as calculated from infrared spectra. (S2 1-287).

The research on accessibility to complexing agents of stable cotton cellulose derivatives has been extended to several other types of formaldehyde-crosslinked cottons. The order of decreasing accessibility of six types of formaldehyde-crosslinked cottons to selected swelling agents has been established. For 12.7% sodium hydroxide (below mercerizing strength) or Triton B the order is as follows: Form W, Form FF, Form W', Form F, Form D, Form V. For more concentrated sodium hydroxides (20.3% and 29.3%) and ethylenediamine which penetrate the crystalline regions more readily, the order changes to: FF, W, V, F, W', D. Basic information of this kind will facilitate advances in chemical finishing research. (S2 1-280).

A wide angle X-ray diffraction technique was developed for evaluating the effect of various swelling agents upon the crystalline structure of cotton cellulose while the cotton is still in contact with the liquid. This new technique has indicated that most of the commonly used swelling agents, except for those that are known to transform cellulose to a different crystal lattice form, have little or no effect on the crystalline regions and predominantly affect the amorphous regions. Previously noted changes in the conventional X-ray diffraction pattern of cotton cellulose as a function of

humidity have been demonstrated to be due to strain and distortion of the crystal lattice which are increased by desiccation. (S2 1-276).

Investigation of the fluorescence spectra of native and modified cottons was continued. Experiments with several types of chemically modified cottons showed that the environmental conditions under which cottons are stored can cause considerable variation in their fluorescence spectra. For some samples, differences in storage conditions resulted in the shifting of excitation or emission maxima in the spectra by as much as 15 millimicrons. Because of this, the interpretation of fluorescence spectra may be difficult; it will be necessary to give careful attention to the storage and history of the modified cottons. Information obtained on the mechanisms of oxidative processes by means of oxyluminescence indicates that further study of this phenomenon would be of value in cotton utilization research. (S2 1-264).

Nuclear magnetic resonance (NMR) spectra of cellulose acetate, ethyl hydroxyethyl cellulose, carboxymethyl cellulose, cellobiose octaacetate, carboxymethyl cellobiose, and the 1:1 adduct of DMEU with ethylene glycol have been obtained. Through the study of such compounds by NMR and other spectroscopic techniques, it should be possible to obtain information useful in elucidating the chemical structure of chemically modified cottons. Also, research on the acetylation of methyl- α -D-glucoside (a model compound of cellulose) has resulted in the synthesis of a mixture of two monoacetates whose NMR spectral properties are currently being investigated to ascertain the position of reaction. Findings from this research may prove useful in interpreting the NMR spectra of cellulose derivatives. (S2 1-268).

In basic studies of the relationship between the spectral properties of selected chemicals and their suitability for improving cotton's properties, alkylene bisamides and biscarbamates and bisformamides, which are used as starting materials in the preparation of crosslinking agents for cotton, were investigated. Intramolecular hydrogen bonding was found to be important in determining the relative conformational stabilities of methylene bisformamide but was relatively unimportant in the other compounds studied. The rotational forms of the two amide groups have been shown to be interdependent in methylene and ethylene bisformamide. The amide moieties in the bisacetamide all exist predominantly in the trans form. No evidence of hindered rotation was found in the spectra of the biscarbamates. Research on diacetyl piperazine and diformyl piperazine is now in progress. The information being obtained will provide reference spectra of the various compounds for future work on finishing agents and will make possible the necessary correlation between group interactions and chemical shift and/or spin coupling patterns. (S2 1-291).

Results of initial investigations of native and modified cottons by wide line NMR spectroscopy appear promising. Preliminary experiments indicate that the disappearance of the proton signal from the wide line spectrum of cotton as air saturated with heavy water (deuterium oxide) vapor is passed over the sample will be valuable in determining the accessibility of hydrogen in

cotton. Also, studies of a wide range of cottons treated with formaldehyde by the Form V and Form D processes indicate changes in the ratio of mobile and immobile protons with treatment. The methods under investigation should provide insight into the structural changes that cotton undergoes during crosslinking processes. (S2 1-309).

Continued research on various types of partially etherified cottons has provided further knowledge of sites of reaction and distributions of substituents in the glucopyranosyl unit of the cellulose as a function of variables such as reagent and process of reaction. The distribution of allyl substituents (reaction with sodium allyl sulfate) has been clarified to be 0.7 : 0.2 : 1.0 in the 2-O-, 3-O-, and 6-O-positions. Significant differences in distributions of methylsulfonyl ethyl substituents result when the reagent or process of reaction are changed. Further study of the reactions of methyl vinyl sulfone and its precursors with cotton cellulose has established that it is possible with a single reagent to obtain ratios of 2-O- to 6-O- substitution ranging from 0.14:1.0 to 0.8:1.0 by employing various conditions of reaction ranging from equilibrium-controlled to rate-controlled. In the reaction of diethylaminoethyl chloride with cotton, it has been found that the ratios of substitution at these two positions can be varied from 1.27:1.0 to 2.5:1.0 by changing the reaction medium (aqueous vs non-aqueous). Thus, true control of chemical reactions of cotton to achieve optimum performance properties appears to be a reasonable potential outcome of continued studies. (S2 1-214(Rev.)).

Cotton crosslinked with formaldehyde at elevated temperatures in the absence of catalyst has been found to exhibit a greater number of effective crosslinks at a specific level of formaldehyde, as measured by sol-gel analysis, than that crosslinked in the presence of catalyst. The effectiveness of the crosslinking reaction on both cotton cellulose and amorphous cellulose (ball-milled) decreases with the presence of catalyst. The technique of gel permeation chromatography employed as a tool for measuring molecular structural parameters of the cellulose polymer is being automated. Thin-layer chromatographic techniques, which would be more rapid and use smaller quantities of sample material, are also being evaluated to determine their utility for routine comparison of samples produced by a wide variety of modifying reactions. Oligosaccharides of high molecular weights (800 to 2000) are being prepared; they will be used to study the range of permeability of celluloses to compounds of molecular weights higher than those of the available sugars previously used. (S2 1-299).

In a P. L. 480 project at the Swiss Federal Institute of Technology, Zurich, Switzerland, a study is being conducted of the chemistry and structural nature of the bonds formed between formaldehyde and cellulose in formaldehyde-treated cottons. Means have been developed for introducing high levels of formaldehyde into cotton, and improved analytical methods for determination of formaldehyde in methylated, formaldehyde modified cottons have been devised and their accuracy and reliability established. Mono-, di-, and trimethylated glucoses have been synthesized and identified, and a procedure

for methylating formaldehyde-modified cotton which does not degrade the oxymethylene linkage has been devised. Gas chromatographic procedures are being applied for the separation and identification of methyl glucoses (in the form of their glucitol derivatives) prepared from the modified cottons, to aid in elucidation of the structure of formaldehyde-cellulose crosslinks. Since the formaldehyde crosslink is widely used in treating cotton fabrics to confer wash-wear properties, the basic data obtained in this project is expected to provide information that can be translated into practical use in devising improved cotton products. (UR-E27-(20)-2).

A study of chemical modifications of cotton fabrics involving control of lateral molecular order and crosslink distribution, conducted under a P. L. 480 project at the Shirley Institute, Manchester, England, is now getting well underway. Methods have been developed for introducing crosslinks into selected locations in the cotton cellulose structure through the use of methyl substituents to block certain positions in the molecule from undergoing crosslinking reactions. Preliminary data suggest that the strength of the ordered regions of cotton cellulose entirely determines the tear strength of the fabric. Methoxyl distribution studies are yielding information on the areas within cellulose which are rendered accessible by water swelling. Basic information obtained in this research is expected to be of value in developing better finishes for cotton fabrics through ability to control and direct sites of attachment of crosslinking reagent. (UR-E29-(20)-78).

A fundamental investigation of the effect of swelling and stretching treatments on the fine structure and mechanical properties of cotton fibers is now nearing termination under a P. L. 480 grant at the Ahmedabad Textile Industry's Research Association (ATIRA), in India. The effect on fiber fine structure, as revealed by X-ray, microscopic and modulus measurements, of swelling fibers under tension with agents such as solutions of sodium hydroxide, ethylene diamine, and zinc chloride has been studied. Small angle X-ray data, before correction for smearing effects, show the same intensity of pattern regardless of swelling history, indicating that the larger voids undergo no change in size due to swelling and stretching treatments. Orientation is considerably more decisive than crystallinity in determining the elastic modulus of cotton fibers. The information obtained in the investigation is expected to be useful in the selection of treatments to improve the mechanical behavior of cotton products. (UR-A7-(20)-19).

In a P. L. 480 project at the Shirley Institute, Manchester, England, a study is underway on the effect of caustic soda and other swelling agents on the fine structure of cotton. The first phase of the project, in which a comprehensive and critical survey of the literature on the swelling of cotton was made, has been completed and the survey published as Shirley Institute Pamphlet No. 93, 247 pp. The publication has been widely disseminated to cotton and cellulose researchers, both in the U. S. and abroad. Its enthusiastic reception gives ample evidence of the value of this work to scientists in the field of swelling and in the practical

implications of swelling in cotton processing. Work is now well underway in the second phase of the project, which is designed to fill gaps in the literature on swelling made apparent in the survey. (UR-E29-(20)-65).

5. Relationship of Gross Structure of Cotton to Behavior of the Fibers in Textile Structures. In grant research at Georgia Tech Research Institute to elucidate the role of fiber morphology in frictional behavior, an instrument and method have been developed for characterizing the surface frictions between single cotton fibers. Surface characteristics of cottons have been found to alter the coefficient of friction between the fibers. Fibers with different characteristics, such as scoured, mercerized, and untreated cottons, have shown characteristic slip-stick behaviors which can be readily observed from the instrument curve. The slip-stick profiles for cottons differ appreciably from those of the manmade fibers, and differences between the profiles of the scoured, mercerized and the untreated cottons also can be detected. It has also been found possible to discriminate between cottons from different stages of mechanical processing, and cottons before and after heating. The measurements indicate only small changes in friction until heat-drying temperatures are above 120°C. Friction between fibers is higher for the slack fibers than for the fibers in tension. The character of the friction curve, peaks and frequencies, is equally as informative about friction as the actual values of kinetic and static frictions. (S2 1-248(Gr.)).

Experimental and analytical investigations of the role of friction in fiber mobility in deformed yarn structures were undertaken in grant research at Massachusetts Institute of Technology. It was found that both bending and recovery are strongly influenced by the magnitude of the frictional effects between fibers, and these effects continue to operate even if the fibers are permanently deformed. The magnitude of the slip required to reach the condition at which individual fibers strain or buckle is dependent on the degree of twist of the yarn. A device developed for measuring the bending moment of fabrics as a function of curvature has aroused considerable interest and is being considered for manufacture by a commercial instrument firm. The research under this grant has placed the mechanics of fabric deformation on a sound theoretical basis. (S2 1-237(Gr.)).

Research has continued under a P. L. 480 grant at the Juan de la Cierva School of Technical Investigations, Barcelona, Spain, to devise means for the measurement of "total hairiness" of cotton yarns and to determine mechanical factors that contribute to the formation of this phenomenon in the spinning of cotton. It is thought that higher spinning speeds cause increased "hairiness," a fuzzy condition resulting from the protrusion of fiber ends from the body of the yarn. Since for many uses yarns are singed to remove this hairiness, the amount of fibers so removed could have important economic implications. An improved transistorized prototype electronic apparatus to measure and record the "total hairiness" of cotton yarns is in the final stages of completion. In a second phase of work, now getting underway, the effect of spinning variables, such as spindle speeds,

ring diameter, roll speeds, pressure and hardness, on the degree of hairiness of the yarns spun will be evaluated. Delineation of the factors causing yarn hairiness is expected to be useful in that it will permit machine adjustments to be made in processing cotton to minimize the formation of hairiness in yarns. (UR-E25-(20)-31).

6. Investigation of Factors Influencing Comfort in Cotton Textiles. In completed contract research at Harris Research Laboratories, Inc., the decrease in subjective comfort ratings of cotton shirtings treated with resins to impart easy-care properties has been shown to be strongly related to the flexural rigidity of the fabrics and the change of this property with increasing moisture content. Within the range of shirting materials suitable for apparel, the most acceptable fabrics, in terms of subjective comfort sensations, are those which have a moderate stiffness. Both soft and harsh fabrics give rise to a lower level of comfort under mild heat stress. It has been found that napping of fabrics treated with high levels of resin improves the subjective comfort response of the shirtings made from these materials. (S2 1-241(C)).

7. Development of New and Improved Methods and Instruments for Measuring the Physical Properties of Cotton and Its Products. In research on methods of appraising abrasive damage on all-cotton wash-wear textiles, further evaluation of the recently developed Accelerotor edge abrasion test showed that it can provide essentially the same information as obtained from wash-and-tumble-dry tests and in about one-twelfth the time. Appraisals of abrasive damage on durably pressed trouser cuffs by these two approaches agree well when newer washers are used in the wash tests; older washers give greater abrasive damage and less reliable data. Evaluations of durably pressed trouser cuffs after wash-and-tumble-dry cycles indicated that visual evaluation and evaluation based on breaking strength loss lead to different conclusions in the testing of a given specimen. On DMEU-treated twill fabrics, there was fair correlation between Accelerotor edge abrasion and tearing strength retention, but poor correlation between either of these variables and flex abrasion. (S2 1-275).

Although the final design of an instrument for counting neps has been completed by the contractor (Stanford Research Institute), construction and evaluation of the instrument are being delayed by difficulty in obtaining delivery of vital optical and high-precision machine parts. The instrument will use an optical system for nep detection combined with separation of the neps from the masses of cotton fiber by means of high-speed airflow. A conventional photodetector system will count the neps thus removed. If a satisfactory method and instrument for counting neps can be developed, this will aid considerably in improving textile processing techniques and equipment. (S2 1-229(C)).

The contractor (Stanford Research Institute) has constructed a versatile fiber mounting device for the light scattering part of the instrument being

developed for determining length, length distribution, and diameter of cotton fibers. Experiments have indicated that the light scattering pattern of fibers of varying diameters is dependent on a single parameter--the cross section for scattering at 90° of unpolarized incident light. Further investigations are in progress to determine if this parameter is a suitable measure of fiber diameter. Recent studies of the light-scattering field due to fibers at scattering angles near 360° (or 0°) have confirmed the existence of a scattering lobe lying in a plane perpendicular to the fiber and extending ten degrees on each side of this plane. These results indicate that the aperture of the scattered-light detection system should be as large as is optically practical, but at least 20° , in order that the response be insensitive to small deviations in fiber attitude. (S2 1-266(C)).

An investigation of the mathematical and theoretical aspects of the relationship between the fiber length distribution of cotton specimens before and after sample preparation is continuing under a P. L. 480 project at the Lodz Polytechnic College, Lodz, Poland. Work is underway to establish the change in relationships between the length distributions of cotton fibers resulting from actions of mechanical processing steps. Excellent progress has been made in the theoretical approaches, and refinements such as the effect of taper on each end of the cotton fibers have been built into the mathematical model. Comparisons of fiber length distributions obtained by the Fibrograph and by single fiber measurements showed that different values are obtained for the same specimens. This difference is ascribed to the shape of the fibers. Developments under the project will be of exclusive benefit to cotton and should permit more sophisticated analyses of the effects of various processing stages on the fiber length distribution of cottons. (UR-E21-(20)-27).

B. Chemical and Physical Investigations to Improve Products

1. Exploratory Chemical Modification and Finishing of Cotton. Exploratory investigations of new ethers and thioethers of cotton cellulose have proceeded along several lines. Chlorocellulose yarn has been used as an intermediate in the preparation of several new amines and thioethers. Mercerized yarn has been found to react with triphenylsulfonium chloride and methallyl chloride. Phosphorylated cotton obtained by the rapid and inexpensive process of curing fabric with sodium acid phosphate has proven very active for graft polymerization of acrylic monomers on cotton. Of particular importance, N-methylolacrylamide, a cellulose-reactive agent, has been used in conjunction with various individual alkyl acrylates to form copolymer grafts on cotton which can be given deferred cures to crosslink the cellulose and impart wash-wear properties. Heating of cotton fabric with 27% sodium methyl sulfate - 15% sodium hydroxide has proven to be a simple, rapid and efficient method of methylation. Of several organic dihalides investigated for crosslinking of cotton cellulose in the presence of alkali 2,2-dichloroethanol appears to be the most active. (S2 1-283).

Cooperative research with the Cotton Producers Institute on reversible chemical reactions potentially suitable for reversible crosslinking and creasing of cotton was continued. A rapid method was developed for measuring the rate of thermal dissociation of potentially reversible crosslinking agents for cotton cellulose. Data were obtained on the kinetics, equilibria, and rates of reversibility of: (1) the attachment of the mixed Diels-Alder adduct of cyclopentadiene and cyclopentadienedicarboxylic acid to cotton, and (2) the uncatalyzed thermal dissociation of malonic esters with isoamyl alcohol. Slower reaction rates are encountered in the cotton environment than anticipated on the basis of results with systems of model compounds. Wrinkle recovery and permanent creases were introduced into cotton fabric esterified with dicyclopentadienemonocarboxylic acid when the esterified fabric was heated at 150°C. Permanently coiled yarn was also made by this basic process. By heating cotton with dimethylcyclopentanetetracarboxylate, a fabric exhibiting increased conditioned wrinkle recovery and thermal creaseability was produced. Other cotton polycarboxylates, particularly cotton mellitate and cotton pyromellitate, exhibited good thermal creaseability at relatively mild conditions (150°C). (S2 1-258, S2 1-258 (Rev.)).

Investigations of the thermal transesterification of cellulose ester linkages have demonstrated that cotton treated with either cyclopentanetetracarboxylic acid or mellitic acid exhibits interesting reversible crosslinks as evidenced by the introduction of creases into the wrinkle-resistant fabric. The fabrics possess good wash-wear ratings, decreased tear and breaking strengths, and the creases, which are durable to laundering, can be ironed out under normal conditions. Reversible-creasing studies of dialkylamino- and methoxyl-activated crosslinked carbamate derivatives of cotton indicate that the activation to thermally break the carbamate linkage in turn deactivates the thermal formation of the carbamate linkage, suggesting that some "balanced activation" is required to produce cotton fabric with thermally reversible creasing properties. (S2 1-261; S2 1-261(Rev.)).

Recent grant research at Textile Research Institute has shown that diester crosslinking does not improve the resilience of partially saponified, benzoylated cotton. In the case of treatment with sebacyl chloride, the fraction of treating agent involved in crosslinking to introduce sebacyl crosslinks, as opposed to monosubstitution, increases as the extent of saponification is increased. Other results indicate that esterification, crosslinking, saponification, in that order, may be the best route for obtaining cotton fabrics with an optimum combination of resiliency and thermoplasticity through benzoylation and treatment with trimethylol melamine. A Schotten-Baumann procedure using benzoyl chloride and aqueous sodium hydroxide has been found to provide a practical method for the benzoylation of cotton fabric. A new thermoplasticity test has been developed: advantages over the conventional fabric folding test are that it is more sensitive and precise and can be performed on yarn or filament samples. (S2 1-240(Gr.)).

In further research on reactions between cotton cellulose and heterocyclic compounds, it has been found that many epoxides which did not react with unmodified cottons, even in the presence of added bases as catalysts, do react with cottons modified to contain basic groups (for example, aminized cottons). Various aminized cottons have satisfactorily catalyzed the opening of epoxide rings in a number of epoxides to bring about their reaction with the aminized cottons without use of external catalysts. Advances have been made in elucidating the mechanism of ring opening of epichlorohydrin in the presence of amines. The reactions of epichlorohydrin, its thioanalog, and other heterocyclic compounds with cotton and with chemically modified cottons have been investigated. (S2 1-282).

Several chemical agents investigated in cooperative research with the International Lead Zinc Research Organization are potentially useful for producing rot-resistant cotton products without imparting color. N-(Tributylplumbyl)imidazole, zinc pyridinethiol N-oxide, triphenyllead laurate and thioethyltriphenyllead, when used alone, imparted good rot resistance but did not improve the weatherability of cotton fabric. However, application of a resin binder and rutile titanium dioxide in combination with each of the compounds enhanced the weatherability of the fabric. In other work, alkali-soluble cotton with relatively high strength retention in the dry state was produced by oxidation of cotton with lead tetraacetate. (S2 1-292).

In exploratory research on the chemical modification of cotton fabrics using reagents in the form of fogs or aerosols, the impracticability of stationary, multiple pneumatic nozzle spraying assemblies for quality textile finishing has been demonstrated. However, a new machine for the continuous application of spray treatments to cotton fabrics has been designed. It should be suitable for fog as well as vapor treatments. Either a reciprocating spinning disk nozzle or a new type centrifugal applicator or fogger will be used. (S2 1-247).

The contractor (Gagliardi Research Corporation) has completed the research investigations of the chemical modification of cotton through treatments with reagents in the vapor phase. In recent experiments, the contractor has found that ultraviolet light to 2600 Angstroms, the shortest wavelength employed, catalyzes certain vapor phase reactions of cotton, giving add-ons which are proportional to the length of time of exposure to the light. Cotton stock, roving, yarn and fabric all exhibit essentially equal reactivity to reagents in the vapor phase. However, a substantial improvement usually results when the cotton has been scoured prior to exposure to the vapors. There is intense commercial interest in vapor phase finishing of cotton, and at least one manufacturer has undertaken the design of a prototype reactor. (S2 1-231(C)).

Further exploratory research on the use of polymers in conjunction with crosslinking agents to improve resistance to edge abrasion in wash-wear cottons has demonstrated that the type of fabric to which the treatments are

applied is an important factor in abrasion performance. Treatment of a soft twill with a polyurethane and crosslinking agent resulted in trouser cuffs with high durable-press appearance, good hand, and excellent abrasion resistance in laundry-wash tests; use of heavy (stiffer) twills led to cuffs with poor abrasion performance. (S2 1-260).

Work has been initiated by the contractor (Bjorksten Research Laboratories) to investigate the effect of resin thermoplasticity or thermosettability on the resistance of treated cotton fabrics to abrasion. Fabrics prepared by grafting an acrylate or methacrylate monomer to the cotton and then treating with a crosslinking resin have been evaluated. The results showed that grafting of diethylaminoethyl acrylate to cotton twill fabrics prior to crosslinking with a urea formaldehyde resin can extend the fabric's abrasion life to 88% of that of an untreated control. The response of fabrics cross-linked with a melamine resin was considerably less satisfactory. Attempts to utilize glycidyl acrylate and several crosslinkable acrylic polymers were not successful. Other agents and types of fabrics will be investigated. (S2 1-290(C)).

Contract research is in progress at New York University to investigate polymer encapsulation of cotton fibers as a means of providing new and useful products. The contractor has conducted exploratory experiments on two approaches for accomplishing the vapor phase encapsulation of cotton fibers: (1) vapor phase addition polymerizations of selected vinyl-type monomers on suitably catalyzed cotton fibers, and (2) interfacial vapor-liquid phase condensation polymerization of polyamides on the surface of cotton fibers. Suitable reaction conditions and processing techniques are being sought. (S2 1-293(C)).

In contract work at Southern Research Institute, reactants containing quaternary ammonium groups have proven to be the most effective compounds for producing wash-wear cotton fabrics with high moisture absorptivity. Their function is at least partially that of swelling agents during cross-linking; there is little evidence of effective hydrophilic activity. Cotton fabrics treated with the N-methylol derivatives of dimethylaminoethyltriazone and of the triazone's quaternary (methyl iodide) derivative had high moisture regain and good dry crease recovery. Various quaternary ammonium salts of the type $R(CH_3)_3NCl$, when added to a pad bath containing a crosslinking agent and catalyst, produced fabrics with relatively high moisture regain and conditioned crease recovery angles. (S2 1-239(C)).

Means for increasing the drying rate of wash-wear cotton fabrics have been found in contract research at Harris Research Laboratories, Inc. Good results were obtained when small amounts of Zepel B, a fluorochemical additive, were used in conjunction with a wet fixation wash-wear treatment and with conventional pad-dry-cure processes. As a consequence of these treatments, the cotton fabrics exhibited greatly decreased water retention when they were centrifuged (spun) in a washing machine. Also, it has been found that the inclusion of 1% perfluorooctanoic acid in the first treating bath

of the National Cotton Council's wet fixation, durable-press treatment decreases by one-third the water content remaining in treated fabric after centrifugation. Permel OE, a polyfluoroalkyl melamine, has proven to be an effective extender for Zepel B in rapid-drying finishes for wash-wear cottons. N-Methylolstearamide also shows some promise. By using extenders of lower price than the fluorochemical additives, processing costs will be reduced. (S2 1-243(C)).

Certain aziridinyl phosphorus compounds have been used as reagents to confer wash-wear and flameproof properties to cotton fabrics in chemical finishing treatments. Research is being conducted under a P. L. 480 project at the Hebrew University of Jerusalem, in Israel, to synthesize and characterize new compounds of this type. A literature survey has been made of the known compounds of this type and sixteen new derivatives have been prepared and characterized. Samples are being provided for screening tests to determine the potential of some of these for utility as improved flameproofing and wash-wear finishing agents. Basic information obtained in this study is expected to be useful in a search for nonyellowing flameproofing treatments applicable to light colored and lightweight cotton apparel and household cotton fabrics. (UR-A10-(20)-56).

2. Chemical Reactions Initiated in Cotton Cellulose and Chemically Modified Cotton by High-Energy Radiation, Light, and Heat. In continuing research studies of radiation grafting of vinyl polymers to cotton, grafted polyacrylonitrile and polystyrene have thus far produced the most interesting changes in fabric properties. Recent work has shown that radiation-grafting of polyacrylonitrile onto cotton to a level of 20% by various techniques produces grafted side chains of a very wide range of lengths (molecular weights - 33,000 to 1,200,000). The length of side chain depends upon a number of factors, such as method of initiation, solvent system employed, and radiation dosage. The fabrics with the longest grafted side chains were most similar in properties and showed the most improvements in properties, particularly increases in flat and flex abrasion resistances and elongation-at-break. Investigations of the distribution of a series of grafted alkyl methacrylates in the cotton fiber structure have indicated that layer opening is produced by the grafting reaction, and this effect is dependent upon the molecular size of the alkyl methacrylate being grafted. (S2 1-195(Rev.)).

Significant contributions toward understanding the free radical chemistry of cotton in reactions initiated by heat, light, chemical action, and ionizing radiation have been made. It has been clearly demonstrated that the localization and transfer of energy deposited within the cotton cellulose molecule from these sources of energy can be directed through chemical and physical modification of the cotton cellulose fiber. This knowledge, which makes possible a more definitive control of desired reactions, has already resulted in the laboratory-scale preparation of weather-resistant cotton products, radiation-resistant cotton products, and cotton products with improved flex and flat abrasion resistances. (S2 1-270).

3. Mechanisms, Rates and Catalysis of Reactions of Cotton Cellulose and of Chemically Modified Cotton. Information has been obtained concerning rates of reaction and activation parameters for the reactions under like catalysis between cotton cellulose and several N-methylolurea derivatives currently employed in delayed-cure processes, as well as some closely related compounds. The role of the metal salt catalyst in such reactions has been more clearly delineated through characterization of the complexes formed between selected metal ion salts and cyclic propyleneurea, and between the same salts and cyclic ethyleneurea. The rates of the pseudo first-order reactions of several agents with cotton for a given catalyst ranked as follows: dimethylolpropyleneurea = dimethylolethyleneurea > dihydroxyethyleneurea \approx dimethyloldihydroxyethyleneurea. In the case of dimethyldihydroxyethyleneurea, the reaction was pseudo first order only with zinc salt catalysts and zero order with magnesium salt catalysts. These fundamental studies are leading to a better understanding of mechanisms of cellulose etherifications. (S2 1-277).

In research on mechanisms involved in producing dry and wet crease-resistant cottons by partial esterification with derivatives of long-chain monobasic acids, various novel methods of esterification with selected long-chain fatty acids are being investigated. Long-chain groups such as the stearyl and oleoyl have been introduced into the cellulose matrix via ester bonds. The wide variation in fabric properties such as crease recovery and resistance to abrasion with mode of esterification indicates that the site of the chemical modification of cotton as well as the type of bonding will affect final fabric properties. In future work, the stearyl and oleoyl groups will be bound to cotton by other groups present in chemically modified cotton or by simultaneous reaction of a polyfunctional reagent with a fatty acid derivative and with cotton. (S2 1-294).

A study of the oxidation of cotton and crosslinked cotton by hypochlorite, hypobromite, and other agents commonly used in bleaching of cotton products is being conducted at the Institute for Fibres and Forest Products of the Ministry of Commerce and Industry of the State of Israel, under a project that is an outgrowth of work conducted under an earlier P. L. 480 project, UR-A10-(20)-4. Basic information is being developed concerning the conditions governing the oxidation, degradation, and yellowing effects that occur on mild oxidation of cottons treated with crosslinking agents commonly used in the easy-care finishing of cotton fabrics. The studies have shown that crosslinking stabilizes cotton to oxidation, and that the effect holds true for various types of crosslinking agents. Oxidation of cotton by bromine preferentially forms ketone groups, an effect that may be useful in the modification of cotton to produce new end-use properties. Functional groups formed during oxidation of crosslinked cottons also give some indication of the sites of attachment of the crosslinking agent on the cellulose molecule and may be useful in determining the average distance between crosslinks. The information obtained in this research is expected to be useful in improving the characteristics of cottons, especially fabrics treated for easy-care properties for various end uses. (UR-A10-(20)-50).

4. Investigations of Soiling and Soil Removal from Cotton Textiles. The contractor (Harris Research Laboratories, Inc.) has developed additional fundamental information on the effect of the soiling environment on the soiling tendency of a series of cotton finishes. It was found that the nature of the chemical finish on treated cotton fabrics has a large effect on both surface potential and surface resistivity of the fabrics. There is some indication that in drycleaning solvent or in aqueous systems fabrics having high positive or negative surface potentials have high levels of soiling. Surface resistivity appears to be influenced even more by the type of chemical modification (esterification) than by the nature of the finish. These basic findings should prove useful in the development of new soil-resistant finishes and possibly in techniques employed for cleaning fabrics. (S2 1-223(C)).

Grant research at the University of Arizona has indicated that electron probe studies of many soiled fiber surfaces can be made when the surfaces have been covered with an ultrathin film of platinum which prevents destruction of the cotton but permits identification of the composition of the soil. The electron probe techniques can determine the chemical nature of the soil particles in situ as well as demonstrate the effects of modified and unmodified fiber surface structure on entrapment of soil particles. A cold incineration (oxidation by activated oxygen) technique developed may be used for those specimens in which the thin platinum layer is an interfering factor. This technique will permit identification of soil particles on cotton samples by electron probe examination and electron diffraction analysis. Information obtained by this approach is being compared with that obtained when replicas of the samples are studied by the electron probe and electron diffraction methods. (S2 1-238(Gr.)).

5. Exploratory Physical Investigations of Native and Modified Cotton. Investigation of the tensile recovery behavior of cotton yarns and fabrics under different conditions and methods of testing has continued. Two approaches for increasing the efficiency of tensile recovery measurements have been found: (1) use of Instron integrator readings instead of chart measurements, and (2) use of stress decay measurements. A comparison of recoverable energy as determined by Instron integrator readings with tensile strain recovery as determined by chart measurements gave a high correlation coefficient, 0.986. It should be possible, therefore, to obtain comparable values by use of the integrator and at a considerable saving in time. Stress-decay measurements could provide useful recovery data with a possible reduction in the total number of specimens required for testing. Studies of several types of durable-press fabrics indicate that tensile and related properties can be useful in helping to predict wear life as determined by wash-and-tumble-dry tests. However, for the results to be most meaningful, comparisons should be restricted to fabrics of similar weight and construction. Fabrics that showed damage after a small number of wash cycles also were exceptionally poor in one or more of the physical properties. (S2 1-285).

Further research was conducted to study the relationship of fabric durability to properties of the untreated cottons. Durable-press treatments applied to comparable balance structure fabrics of approximately printcloth weight made from Pima, Hopi Acala, and Deltapine cottons, whose fiber properties are appreciably different, eliminated differences in abrasion resistance observed in the untreated cottons, except when the cottons were slack mercerized before the resin treatments. In the latter case, there was a marginal but consistent advantage in abrasion resistance for the Pima fabric over those made from the two Upland cottons. The slack mercerization caused a larger increase in abrasion resistance over that of the scoured fabrics than attainable by selecting superior-quality cotton. Results of wash-and-tumble-dry tests on durable-press trouser cuffs made from Pima, Hopi Acala, and Deltapine fabrics of various constructions showed that, in general, the Pima cotton gave the best abrasion resistance performance and Hopi Acala was intermediate. For comparable weight fabrics, the basket weave structures had greater abrasion resistance than the printcloth structures. Fabrics that were slack mercerized prior to the resin treatment gave consistently better performance than comparable scoured, resin-treated fabrics. No large advantages in recovery from stretching could be attributed to any one of these varieties of cotton over the others. (S2 1-272).

In further research to determine the simultaneous effect of pertinent fiber properties and combinations of fiber properties on yarn properties and spinning performance, it was found that no individual yarn property correlated highly with end breakage in spinning, based on simple correlation analysis. Another finding is that the amount of twist required by a cotton yarn to yield maximum breaking strength depends directly on the fineness and inversely on fiber length and toughness. It was established that the mathematical formula for predicting yarn twist for maximum yarn strength can be used within limits of about .15 twist multiplier units. A unique graphical method was developed which can be used to obtain approximations of Suter-Webb array information from Digital Fibrograph data. Another important result of the research is a formula for predicting single yarn strength from the fiber bundle tenacity measured at 1/8" gage length, effective weight, yarn number, and bundle elongation. It was also shown that the percentage of fiber strength translated into yarn strength is dependent on yarn number and twist, and on fiber bundle and yarn strength measuring techniques. (S2 1-207).

Completed research by the contractor (Macrosonics Corporation) on the ultrasonic treatment of cotton fibers in liquid media indicates the possibility of utilizing acoustic energy in producing bulked cotton yarns and in irradiating short fibers for use in making paper. High-power ultrasonic irradiation does not appear to have merit as a tool for trash removal from raw cotton. (S2 1-222(C)).

A basic investigation of the behavior of cotton fibers when subjected to aerodynamic forces is now nearing completion in a P. L. 480 project at the

Chalmers University of Technology, Gothenburg, Sweden. High-speed motion picture photography has been employed in a specially designed apparatus to visualize and study the action of individualized cotton fibers when borne by airstreams of different velocities. Conditions and designs were sought which would cause the fibers to become parallelized through aerodynamic forces exerted by the flowing airstream. Early studies using relatively low air velocities provided much useful information concerning the behavior of cotton fibers under various conditions. However, not until the later stages of the investigation, when high velocity airstreams were studied, was a substantial degree of fiber alignment achieved. Fundamental information concerning the conditions required to separate and parallelize cotton fibers aerodynamically is a prerequisite for the development of new and unorthodox methods for processing cotton, such as direct fiber-to-yarn open-end spinning by several proposed procedures. (UR-E26-(20)-6).

An investigation of means to minimize fiber hooked ends in cotton card and drawing slivers is in progress under a P. L. 480 project at the Ahmedabad Textile Industry's Research Association in India. End breakage in the processing of cotton is related to the presence of hooked ends in the fibers making up sliver. Conventional processing organizations tend to remove hooked ends, but in abridged processes such as direct spinning, the fewer drafting processes between carding and spinning allow more fiber hooks to remain and hence adversely affect spinning efficiency, since fibers with ends hooked do not make their full potential contribution to strength and other characteristics of the yarn assembly. Studies made of the effect of relative speeds of the machine elements in the card, and of the loading of the cylinder and doffer have indicated that increasing the loading decreases the minority hooks but increases the majority hooks. Work is now underway to clarify the relationship between fiber hooks and card loading and to study the effects of higher card production rates, which should lead to a reduction of majority hooks. Basic information obtained in the research is expected to be of use in facilitating direct spinning and improved cotton processing through means to minimize the amount of fiber hooks in card or first drawing sliver. (UR-A7-(20)-51).

Research is in progress under a P. L. 480 project at the Juan de la Cierva School of Technical Investigations, Barcelona, Spain, to determine the effect of fiber properties on drafting tenacity during the spinning of cotton and the interrelationship between fiber properties, drafting tenacity, and end breakage. An apparatus developed under an earlier P. L. 480 project at the same institution which permits drafting tenacity to be measured and recorded directly during drafting is being employed as a key instrument in the present work. Early findings concerning the effect of relative drafting speed on drafting force indicate that fiber frictional forces increase with speed. The effect of fiber hooks on drafting force was present when spinning carded yarns, but no directional effect could be detected when spinning combed yarns. A definite correlation has been found between drafting tenacity and the tenacity of yarn to break. Information of the

type being obtained is expected to be useful in the development of abridged, or "short-cut" cotton processing systems. (UR-E25-(20)-42).

A study of the factors that affect drafting capacity, spinning efficiency, and yarn quality of the direct sliver cotton spinning system is being conducted under a P. L. 480 project at the Ahmedabad Textile Industry's Research Association at Ahmedabad, India. Early findings have indicated that the effects of total draft on yarn characteristics obtained with the direct (sliver) spinning system are similar to those obtained with the conventional (roving) spinning system, in that for coarse yarns the effect of total draft is more critical than for medium and fine yarns. Effect of spinning draft on yarn strength is more critical in roving spinning than in sliver spinning. Thus far, the roving-ring frame has out-performed the OMS direct drafting system. The latter, however, is being modernized to obtain a more comparative test. The study will be extended to a wider range of yarn numbers and roving hanks to obtain complete spinning draft curves. Basic information being obtained will have practical applications in the development of more useful abridged spinning systems for cotton. (UR-A7-(20)-85).

The Shri Ram Institute for Industrial Research, in Delhi, India, is conducting a study of the correlation between several important physical properties of cotton apparel fabrics and their performance in actual service tests under a P. L. 480 project now in its early stages. Pilot scale wear tests at a boys' school involving shirts made from fabrics given four different finishing treatments are underway. Limited correlation analysis of the data obtained thus far indicate that both tensile strength and tear strength correlate well with in-service wear. An apparent lack of correlation has been found between in-service wear and several laboratory abrasion tests used. The work will be extended to more complete in-service testing of fabrics in work clothing and in military apparel. The information obtained in this project is expected to fill gaps in existing knowledge of the correlation between the results of test methods related to fabric wear, and actual performance in clothing applications. (UR-A7-(20)-87).

The mechanism of crease formation and recovery in ease-of-care treated cotton fabrics is being investigated in a P. L. 480 project now in its earlier stages at the Swedish Institute for Textile Research, Gothenburg, Sweden. Relationships between crease recovery and environmental factors, both during the creasing and the drying of fabrics, have been illustrated. Tests on fabrics given wash-wear treatments by several chemical methods have shown that recovery as related to water present varies with the degree of water swelling during treatment. Work is being extended to studies of graft polymerization on set and unset cotton fabrics in a two-step reaction involving single end reaction of N-methylol groups with cotton cellulose followed by polymerization of vinyl groups of the N-methylol-acrylamide reagent. A new apparatus designed for recording crease recovery angle versus weight of the sample is being developed for use in the research. The results of the work will afford information needed for the design and treatment of improved easy-care cotton fabrics. (UR-E26-(20)-1).

Research is progressing under a P. L. 480 project at the Shri Ram Institute for Industrial Research, Delhi, India, on an investigation of heat and mass transfer rates and other engineering concepts as related to the drying and curing of resin-treated cotton textiles by countercurrent solid-gas contact systems. The system originally devised has been modified to increase the hold-up of the beads used for the fluidized bed heat transfer medium from 1.5% to 4.0%. Electrostatic problems have been encountered and studies are underway to eliminate this effect. Studies have been made of mass transfer in relation to resin curing on fabric and of heat transfer at various degrees of hold-up in the fluidized bed. Heat transfer comparisons have been made using a conduction system, and a convection system, in addition to the fluid bed system. Fluidized bed techniques provide highly efficient means for effecting heat transfer in many engineering applications. The basic engineering data being obtained in this research is prerequisite to adapting this system to the drying and curing operations in textile processing. If successful, the adaptation of the fluidized bed technique will be of great practical value in increasing efficiency and lowering costs in cotton textile finishing. (UR-A7-(20)-84).

C. Technology--Process and Product Development

1. Improved Procedures for Mechanical Processing of Cotton. In research to determine the interaction of processing variables with yarn properties and end breakage, an exploratory evaluation of drawing frame variables was made and an appropriate range of spinning twist multipliers for the drawing, roving and spinning phases of the research was established. Preliminary results indicate that higher doubling-and-draft at the drawing process produced the greater projected lengths of fibers. High draft at the drawing frame was more efficient than low draft in removing total hooks, doing so primarily by removing trailing hooks. In contrast, low draft appreciably reduced leading hooks. Work is in progress to develop a simpler, more rapid clamping technique for determining projected mean length of fibers and number of hooks in each direction. (S2 1-295).

Research has continued under contract at Auburn Research Foundation, Inc., to determine optimum processing procedures for cottons differing in tensile and elastic properties, and relate these properties to mechanical processing performance and yarn and fabric properties. Laboratory processing performance evaluations of short staple cottons differing in fiber elongation were completed. Generally, the trends found for these cottons were not similar to those found earlier for medium staple cottons. High elongation in the medium staple cottons generally had a more pronounced effect on end breakage in spinning than did high elongation in the short staple cottons, probably because of the inverse relationship between fiber elongation and fiber strength of the latter cottons. For the short staple cottons, fiber bundle elongation and end breakage were not directly related. In most cases with these cottons, the medium fiber elongation cotton spun better than the low elongation cotton; however, the high elongation cotton spun worse than either the low or medium elongation one. Regardless of fiber elongation and spindle

speed levels, end breakage rates were significantly higher when yarn tension was increased. Use of higher yarn tension also resulted in a decrease in yarn break elongation. (S2 1-242(c)).

The research investigations to determine the effect of high production carding on fiber length distribution and fiber hook formation in card sliver and to establish improved drafting procedures required for maximum removal of fiber hooks for carded and combed yarns have been completed. High production carding tests on a 1-3/16" irrigated Acala 1517 cotton showed that increased carding rate caused the minority fiber hooks to increase at a much faster rate than was previously experienced with Deltapine, Lankart, and Acala 4-42 cottons. The majority hooks decreased with increased carding rate, but at a lesser rate than with the other cottons. End breakage rate in spinning for the SRRL drafting directions was less than for other drafting directions. Use of higher carding rates and the SRRL drafting directions both led to reductions in noils being removed at the comber. Evidence was also obtained that the same mechanism that causes minority hooks may cause neps. A special "single ends" down accelerated spinning test has shown that there is an indirect linear relationship between the amount of hooks leading into spinning and the spindle speed. Yarn strength decreased linearly with increases in leading hooks. As the total amount of hooks decreased during drawing, there was improvement in short term sliver uniformity, yarn strength uniformity, and yarn uniformity. Other experiments on selected cottons indicated that time of harvest had no influence on fiber hooks or processing performance. (S2 1-274).

2. New and Improved Mechanical Processing Machinery--Opening Through Carding. Results of mill-scale evaluations of the full-size SRRL Bale-Opener-Blender have been quite encouraging. The machine's relatively trouble-free performance, its blending characteristics, and especially the size and uniformity of tufts it produces have been pleasing to industry. In three independent evaluations, higher quality fabrics have been produced from stock processed by the Blender than from stock processed by standard mill machines. The new machine also reduces processing costs and space requirements. Because of inconsistencies in manual loading and inherent bale variations, manual adjustment of the Blender's feed control is now required. To insure even production from the Blender without attendance, an automatic feed control mechanism will be incorporated into the machine. Several large U. S. manufacturers of textile mill machinery are interested in obtaining a license to manufacture the Blender. There is also considerable interest among foreign machinery manufacturing firms. (S2 1-252).

A full-size unit of the recently developed Lap Drafter, both with and without a specially designed system for feeding well-opened fibers to the carding cylinder, was installed on a textile card and evaluated. Initial results showed that yarn quality decreased as production rate of the card was increased from 10 to 40 pounds per hour. The quality of the yarn will be compared with that of yarn produced from the same cotton using a standard

card. A friction-type feed unit for delivering loose cotton to the Lap Drafter at a uniform rate is also being developed. Based on tests of several materials with a recently developed instrument for determining frictional properties, it appears that a polished, lightly knurled steel surface may be the most suitable for this feed system. The research investigations on feeding multiple laps to the card and on precarding were discontinued because a new commercial machine developed by industry encompasses both of these features. (S2 1-279).

Two experimental units evaluated for improving fiber parallelization and removing short fibers at the card have both improved fiber orientation of the output sliver to approximately that of sliver from the first drawing frame; however, neither unit gave the desired short fiber removal. Results to date with an electrostatic strand-forming mechanism have been encouraging. Within a single, small machine, raw cotton has been successfully converted into a highly oriented, continuous textile strand. However, the production rate is low, and the strand is nonuniform and crude in appearance. Further research is needed to develop an efficient high-production system for feeding individualized fibers to the mechanism. (S2 1-273).

Because of shortage of required personnel, only limited exploratory investigations of basic actions in cotton textile processing equipment by means of high-speed photography have been possible. A high-speed motion picture study was made to determine the relative motion of long and short fibers during drafting, but no conclusive results were obtained. (S2 1-278).

3. Wash-Wear Cotton Textiles and Garments. One phase of research has been concerned with the fixation of polymer formers and crosslinking agents on cotton in such a fashion that the treated fabrics, after curing, possess increased abrasion resistance over that of conventional wrinkle-resistant fabrics. Improvements in formulations and conditions for producing durable-press cotton fabrics by variations of the National Cotton Council's wet-fixation process have been made. Cuffs with improved abrasion resistance as measured by wash-and-dry tests resulted when the wet fixation was allowed to occur at room temperature or in the presence of a swelling agent (tetraglyme) under standard oven-drying conditions. Also, magnesium chloride has been found to be a more satisfactory catalyst than zinc nitrate for the curing step of the process. Synthesis of new, stable crosslinking agents has also been continued. The N-methylol derivatives of ethylidene-bis-3-(2-pyrrolidone) appear to be a particularly promising new crosslinker for cotton and suitable for delayed curing. Fabrics finished with this new agent were equal in performance and durability to those finished with the carbamates, yet the agent released little or no formaldehyde during storage. (S2 1-289).

In further research on new and modified carbamate finishes for deferred-cure processing to yield chlorine-resistant, lightfast cotton fabrics, it has been found that substitution of a methoxyl, hydroxyl, or chloro group on

the ester moiety of dimethylol ethyl carbamate decreases the ease of cure sufficient to yield a chlorine-resistant fabric. Zinc chloride has proven to be an effective catalyst in the treatment of cotton with carbamate agents. A new, easy-cure process for finishing cotton with carbamates has also been discovered. In this process, drying and curing are carried out at low temperatures (60° C. or less) and low concentrations of hydrogen chloride are employed as catalyst. Fabrics finished by the easy-cure process have good wash-wear ratings after either tumble- or drip-drying. Acetylated dimethylol carbamates have proven suitable for conventional or deferred-cure processing of cotton and have the additional advantage of producing less formaldehyde odor in the dried fabric before it is cured. Other work has shown that bromine retention by treatment of carbamate-finished cottons with sodium hypobromite is much greater and more damaging than chlorine retention by similar treatment with hypochlorite. (S2 1-281).

Crosslinking partially swollen cotton is being investigated as a possible method of improving the abrasion resistance of wash-wear cotton fabrics. An increased degree of water swelling in wet crosslinking and preswelling before dry-cure crosslinking did not improve abrasion resistance. Only in isolated instances did preswelling improve the abrasion resistance given by polymeric coatings. Conditioning to modify the reaction of a crosslinking agent with cotton fabric before curing has not increased abrasion resistance. Such conditioning with dry heat in the presence of an additive to restrict crosslinking or with moist heat gave only slight increases in wear life of treated cuffs. New reaction conditions that modify the reaction of crosslinking agents within the cotton fiber will be studied. (S2 1-296).

The fixation of resilient polymer films on cotton textiles to impart wash-wear properties, durable shape, and increased wear life is also under investigation. A durable-press finish has been found which imparts to all-cotton trouser cuffs a laundering abrasion resistance equal to that of a commercial Koratron-treated 85/15 blend of cotton and nylon. An emulsion containing a high molecular weight dimethyl silicone, benzoyl peroxide, and dihydroxydimethylolethyleneurea is applied to the cotton in the absence of cellulose crosslinking catalysts. By using a mixture of two different N-methylol agents (dihydroxydimethylolethyleneurea and trimethylolmelamine) in this system, an improved treatment has been developed. In this modified formulation, only half as much silicone and peroxide are required, which decreases the potential cost of the treatment. The finished textiles have very high wrinkle resistance. Little cellulose crosslinking occurs in the process thus leading to increased retention of strength and abrasion resistance. Two simple procedures have been found for bonding hydrogen peroxide to cotton to produce textiles with oxidant properties. These modified textiles may be useful in graft polymerization or in crosslinking of polymer films on cotton. Crosslinking of acrylate esters of cotton cellulose, either by heat and acid catalysts or by alkaline hydrogen sulfide, has produced fabrics with high wrinkle resistance. (S2 1-300).

In research recently initiated under a new project, the use of film-forming polymers that coat the individual fibers in a fabric structure in combination with covalent crosslinking agents that penetrate the fibers is being investigated as a means of producing abrasion-resistant, durably pressed cotton goods. Through use of long-chain quaternary additives, the abrasion resistance of durable-press cotton cuffs has been increased two- to fourfold over that of comparable cuffs receiving a conventional deferred-cure finish. Further improvement in reducing the stiffness and increasing the abrasion performance of polymer-treated durable-press goods was achieved by warp compaction of sensitized fabrics and by a chemical blending technique using a mixture of polymers. It was demonstrated that the abrasion resistance of durable-press fabric prepared from low grade, short staple cotton can be significantly improved by premercerization. Effort will be continued on these several lines of work. (S2 1-306).

Another approach to durable-press--weaving fabrics from polymer-treated cotton yarns and then applying the finishes--also appears promising. In preliminary experiments, good durable-press appearance and abrasion performance were both achieved with some of the fabrics. The effects of fabric structure and finishing processes on the physical properties and wearability of the fabrics will be determined. (S2 1-308).

The blending of treated and untreated cotton fibers has also been used successfully to improve the abrasion resistance of wash-wear, durably pressed goods. The initial fabrics made with 50/50 blends of untreated and resin-treated cotton fibers proved to have excellent abrasion resistance, as indicated by home laundry evaluations, but suffered from excessive shrinkage after curing. However, recent investigations with a 3/1 twill (7 oz./yd.) containing such a blend of fibers have shown that preshrinking the fabric by either sanforizing or compaction can be used to overcome this shrinkage problem. By use of these preshrinkage treatments, residual shrinkage of the fabric after 10 launderings was less than 2% in the warp and 1% in the filling. Laundering tests indicated that sanforizing apparently gives slightly greater improvement in abrasion resistance than does compaction. The compacted samples showed as much as 13% stretch in the warp direction. As a possible means of improving mechanical processing and reducing cost, the use of unscoured, unbleached rawstock is being studied. (S2 1-286).

Research under a new contract at Fabric Research Laboratories, Inc. will be directed toward the development of methods for improving the dimensional stability of abrasion-resistant, durable-press fabrics made from blends of untreated and resin-treated cotton fibers. The contractor is presently procuring necessary materials and supplies. (S2 1-304(C)).

Significant improvements in abrasion resistance and wash-wear performance of durably pressed cotton goods have been achieved by use of the poly-set process, a two-step process involving controlled polymer deposition and crosslinking with an N-methylol crosslinking agent. Work is currently

underway to obtain further improvements, particularly a higher degree of wrinkle recovery in step one (the polymer deposition step) so as to produce muss- or wrinkle-resistant fabrics with minimum losses in strength properties. (S2 1-311).

A study of cellulose reorganization techniques for preparing stabilized durable-press cotton fabrics is in progress. Application of a cellulose-reactive dye to cotton fabrics woven of premercerized yarns, followed by a deferred-cure treatment with Permafresh 183 in combination with surface polymers, has resulted in durable-press trouser cuffs that exhibit little or no frosting after 31 laundering test cycles. When a vat dye was used in place of the reactive dye in the same treatment, a great deal of the objectionable frosting occurred during this period of laundering. High temperature mercerization will be investigated as a pretreatment for wash-wear finishing and delayed curing of cotton fabrics. (S2 1-310).

The application of zirconyl ammonium carbonate or ammonium stearate to fabric by padding followed by heat decomposition prior to crosslinking has produced fabrics with higher breaking and tearing strength, better flex life, and improved resistance to abrasion in repeated washing and tumble drying cycles. Apparently the hydrophobic materials, zirconium oxide and stearic acid, produced by the heat treatment block the most readily accessible sites in the fiber where initial crosslinking would normally take place. Other promising approaches being studied include mechanical compaction of fabrics in the warp and filling directions and slack mercerization, which impart stretch properties and enable the fabrics to absorb more energy, thus improving abrasion resistance. Durable-press pants cuffs made from slack mercerized, warp compacted fabrics showed less wear after multiple wash- and tumble-dry cycles than did comparable cuffs made from unmercerized, warp compacted fabrics. Filling compaction is expected to have some advantages over warp compaction. (S2 1-288).

Cooperative research with the Cotton Producers' Institute has definitely established that certain fabric structures provide the best resistance to abrasion in durable-press cotton fabrics. In completed laundering tests on durable-press trouser cuffs made from various experimental ply-yarn fabrics, the 3/2, 45° twills wore best, followed in order by the 3/2, 63° twills, sateens, and plain weave fabrics. Fabrics woven from heavier yarns wore better than those of comparable weight woven from lighter yarns. Preliminary results from a completed study of equivalent fabric structures made from singles yarns indicate that the single-yarn fabrics generally exhibited better wear characteristics than the ply-yarn fabrics. One of the single-yarn fabrics, a 3/2, 45° twill, showed no damage after 115 wash-and-tumble-dry cycles. As a consequence of this research, several industrial concerns have requested assistance in redesigning their fabrics for improved durable-press performance. Also, men's all-cotton durable-press seersucker suits tailored from fabric meeting specifications developed in this research have

met with outstanding success in market tests sponsored by the National Cotton Council. (S2 1-254).

Completed contract research at Georgia Tech Research Institute has established that the following three factors contribute 76% of the predilection to pucker in wash-wear cotton products: sewing direction, type of seam, and dimensional compatibility of components being sewed together. Although laundering does intensify some forms of puckering, fabric finish itself is not a major factor. None of the treated threads investigated combined both sewability and improved after-laundering performance. Findings from this research should contribute materially to the development of techniques and countermeasures needed to overcome the seam pucker problem in wash-wear cotton products. (S2 1-228(C)).

4. Weather-Resistant Cotton Fabrics. Cooperative work with the Canvas Products Association International and the Foundation for Cotton Research and Education has resulted in the development of a new process, the "Zirchrome Process," which produces a superior pearl-gray base finish for outdoor cotton fabrics by a chromium oxide-zirconia mineral dyeing. In weathering tests, experimental fabrics treated by the new process proved much more weather-resistant than those treated by the conventional mineral dyeing processes. The zirchrome-finished fabrics, even without a protective coating, retained 90% of their original strength after 24 months, whereas the conventionally treated, uncoated fabrics retained only 40 to 60% of their strength after just 12 months. With the zirchrome process, protective agents such as fungicides, mildew preventatives, and waxes for waterproofing can be applied simultaneously with the mineral dye in a single bath. In contrast, the processes now in general use require up to four baths and numerous processing steps. At least twelve firms are already evaluating the new process, and six of these have produced pilot-plant quantities of zirchrome-finished fabrics. Another recent research development is a simple, one-bath process for the application of water-insoluble organic dyes to cotton fabrics by solubilization of the dyes with zirconium acetate. With this process, brilliant color shades can be produced with only half the concentration of dye required for comparable shades of conventionally dyed fabrics. (S2 1-259).

An excellent colorless weather-resistant finish for outdoor cotton fabrics has also been discovered in the cooperative research. It is a clear-base finish based on methylolmelamine resin and zirconium acetate. Cotton fabrics treated with this finish and coated with a pigmented vinyl coating are still retaining more than 90% of their original strength after 36 months of outdoor weathering in the warm, moist climate of New Orleans, Louisiana. This good performance should make the fabrics very attractive to industry for use in awnings, truck and boat covers, tarpaulins, tentage, and other canvas products. At least three manufacturers are actively considering the new development. In another phase of work, a comprehensive series of experimental weather- and mildew-resistant fire-retardant treated cotton fabrics has been prepared. Evaluations of the fabrics in outdoor weathering tests are in

progress. In other research, the role of air pollution in the accelerated degradation of exposed cotton fabrics has been studied in the laboratory by means of artificially contaminated samples exposed to controlled visible and ultraviolet light sources. There are strong indications that light accelerates the degrading effects of polluted air. (S2 1-256).

The contractor (Texas Woman's University) has finished the final exposure phase of the research study on weather-resistant, water-repellent finishes for cotton fabrics. Testing of samples has been completed and the data are being analyzed. (S2 1-200(C)(Rev.)).

In contract research at Southern Research Institute to develop improved weather-resistant cotton textiles by interfacial and graft polymerization techniques, some improvements in rot and abrasion resistance have been noted in specimens of duck and drill grafted with acrylonitrile, styrene, or methyl methacrylate by a radiation initiation method. Polyurethanes applied by interfacial polymerization were more effective than a similarly applied polyester in improving rot and abrasion resistance. Unfortunately, none of the aforementioned treatments gave any appreciable protection from deterioration by outdoor weathering. Polyacrylonitrile applied to cotton by the ferrous ion-peroxide method concentrated in the lumen of the fiber; however, grafting by the radiation initiation method resulted in a uniform distribution of the polymer throughout the cell wall. (S2 1-245(C)).

Contract research to develop improved coated cotton fabrics with optimum strength-weight characteristics for outdoor uses is in progress at Fabric Research Laboratories. The initial evaluation phase of the investigation has been completed. Data on the performance qualities of the various structures are being obtained and analyzed. (S2 1-244(C)).

5. Soil-Resistant Cotton Textiles. High oil repellency has been imparted to cotton fabrics by large-scale application of the ethyl perfluorooctanoate-ethylenimine finish from either a solvent system or an emulsion system. The treated fabrics have properties comparable to those obtained in smaller scale work. The finish is durable to home laundering and solvent extraction; and the soiling properties of the treated fabrics are similar to those of the untreated, except for somewhat increased soiling with oily soil. Another fluorochemical finish, the THPC-fluoroamine emulsion finish, has also been evaluated on a large scale. It imparts high oil repellency to cotton fabrics and has good durability when cured with gaseous ammonia. Fabrics treated with this finish soil like untreated fabric, but soil removal is slightly inferior. Both of these new oil-repellent finishes are superior to several commercial fluorocarbon finishes in regard to oily soiling and oily-soil redeposition but are slightly inferior to some in aqueous-soiling properties. Preliminary experiments indicate that it will be possible to employ either of these new finishes in conjunction with permanent-press finishes to achieve both wrinkle resistance and oil repellency. (S2 1-297).

6. Flame-Resistant Cotton Textiles, Including Those with Multifunctional Properties. Research is being conducted to develop flame-resistant cottons with improved abrasion resistance and durable-press properties. Light- and medium-weight cotton fabrics were rendered flame resistant by the tetrakis(hydroxymethyl)phosphonium hydroxide (THPOH), ammonia-fixation process with little or no strength loss or change in hand of the fabric. This promising process should be useful for heavyweight fabrics as well. Improved flex abrasion resistance and modest flame resistance were given to cotton fabrics by treatment with low add-ons of THPC-acrylamide-urea polymer. Higher add-ons of the polymer conferred a greater degree of flame and wrinkle resistance, but abrasion resistance was improved to a lesser degree. Durable-press properties and modest flame resistance were imparted by pad-dry-cure treatment with a PNE (allyl ester of phosphonitrilic chloride trimer) emulsion followed by backcoating with a conventional crosslinking resin. When the PNE was applied by surface application to the face of the fabric, there was improvement in abrasion resistance but flame resistance and durable-press properties were not as good. The THPOH and PNE processes will be investigated further. (S2 1-305).

New finishes for imparting flame, wrinkle, stain and abrasion resistance to cotton products are also being investigated. Two new compounds, diethyl 1H, 1H, 2H, 2H-pentadecafluorononyl phosphonate and perfluoroheptyl tris(cyanoethyl)phosphine, were synthesized for use as intermediates to multipurpose finishes for cotton, particularly finishes which impart oil and water repellency as well as flame resistance and wrinkle resistance. Cotton fabrics treated with a newly discovered oil-repellent finish based on a polymer of polyethylenimine and ethyl perfluorooctanoate were found to soil less with oily carbon soil and have less soil redeposition than fabrics treated with two commercial stain-resistant finishes. This new finish was made slightly more durable to laundering by adding APO to the formulation. Polymers of the ethylenimine adducts of acrylonitrile and methylene glutaronitrile have improved the flex abrasion resistance of cotton fabric and might prove useful in durable-press applications. (S2 1-298).

7. Cotton Textiles with Improved Luster. In recent research to develop lustrous wash-wear cotton fabrics with increased strength and durability, it has been found that the losses of flex abrasion resistance normally produced by crosslinking cotton fabric with DMEU resin can be prevented by application of a crosslinked film of a high molecular weight silicone along with the DMEU to fabric woven of slack mercerized, partially restretched yarn. The silicone also greatly increased the degree of wrinkle resistance obtained; the dry and wet crease recoveries (W+F) of the treated fabric were 306° and 294°, respectively. (S2 1-267).

8. Stretch and Bulked Cotton Products. Wear testing of the knitted T-shirts and socks made from resin-treated cotton stretch yarns (produced by the back-twist and false-twist methods) and from control yarns has been completed by the contractor (North Carolina State University), and the data have been analyzed. The stretch items received higher ratings in stretchability and

ability to yield during wearing but lower ratings in shape retention, softness, surface texture, smoothness, moisture retention, and durability. Knitting either of these types of stretch yarns requires machines with 40% greater needle spacing than those normally used for regular cotton yarns of comparable size. (S2 1-197(C)(Rev.)).

Other contract research at North Carolina State University has indicated that it is not practical to produce stretch cotton T-shirts by slack mercerizing a loosely knit T-shirt because of puckering, sewing problems, and differential shrinkage. However, by fabricating T-shirts from slack mercerized knitted cotton fabric, it has proven possible to produce stretch T-shirts with dimensional stability. Experimental stretch socks that are generally satisfactory in appearance, hand, and stretch properties have been made by slack mercerizing oversized, loosely knit cotton socks. The properties of these two types of stretch garments are generally superior to those of the conventional, nonstretch garments. Commercial development of the new products will be dependent upon the economics of the processes. (S2 1-224(C)).

Recent cooperative investigations with the National Cotton Batting Institute, and the Textile Waste Association, the National Cottonseed Products Association, and the Foundation for Cotton Research and Education have resulted in the production of chemically treated cotton batting with improved characteristics. Odor problems in the finished batting have been significantly reduced by incorporation of odor suppressants and masking agents. Progress has been made in developing low-cost flame-retardant treatments which will make Cotton Flote safer for various end-use applications in the automotive, bedding, and furniture industries. Low-grade CCC cottons appear to be suitable for partial replacement of the linters and textile waste normally used in the batting. Twenty-two companies are now licensed to produce Cotton Flote, and at least twelve are either in production or installing the process in their plants. Current production is estimated to be in excess of 4 million pounds per year. The batting is currently being used by two major U. S. automobile manufacturers for cushioning; by another firm for insulation in auto trailers and trailer trucks; by at least two bedding companies for high-grade mattresses; and by a segment of the furniture industry for cushioning applications. Companies in at least fourteen foreign countries have also shown interest in the new product. (S2 1-269; S2 1-269(Rev.)).

9. Insect-Resistant Cotton Bags. Research to develop improved insect-resistant cotton bags for the storage and shipment of food commodities has continued in cooperation with the Stored-Product Insects Research and Development Laboratory at Savannah, Georgia, bag manufacturers, and the Textile Bag Manufacturers Association. Large-scale food storage tests (75,000 pounds of flour and cornmeal in 100-pound bags) are in progress. Cotton bags treated with insect repellent, lined with wax paper, and closed with tape-over seams, and multiwall paper bags treated with the same repellent have given comparable performances after six months' exposure in

the tests. There was only slight insect infestation, and the insecticide contamination of the stored food was within acceptable levels. Current emphasis is on two approaches: (1) various types of lining materials and polymeric coatings to reduce migration of insecticide into the stored food, and (2) additives in the treating formulations to aid retention of insecticide on the cotton. Investigation of improved techniques for fabricating the bags is also contemplated. (S2 1-271).

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AREA 2 - COTTONSEED UTILIZATION - FOOD

Problem. Cottonseed products, including approximately two billion pounds of oil generally derived from the annual domestic production of cottonseed, face increasing competition for markets. For its chief market, edible products, cottonseed oil must compete with other vegetable oils and animal fats. The nation's capacity for producing these oils and fats is so great that supplies can be expected to exceed both domestic and foreign demand for some time to come. Improvements in the quality and utility of cottonseed oil are needed to retain present markets and open new ones for the currently large and possibly greater future production.

Much research is urgently needed on the fungi and toxic fungal metabolites that may develop in cottonseed and its processed products. The mycotoxin problem is a potentially serious one for many agricultural commodities. Also, additional information is urgently needed on the chemical, physical, and biochemical properties of cyclopropene fatty acids in cottonseed and on means of converting them into physiologically inert forms. Recent reports state that cyclopropenoids are potentiating agents to aflatoxins in the production of hepatoma in trout. Another problem is contamination with salmonella, particularly since it may occur at so many stages of processing. Research is urgently needed in these areas.

There is discrimination in the markets against 25% to 50% of the production of cottonseed oil because of the presence of reddish colors that are not removed by conventional commercial refining, bleaching, and deodorizing methods. It is essential that information be developed on the chemistry of the pigments responsible for the off-colors, and that more efficient means be developed to eliminate them and thus upgrade the oils, particularly for use in margarine and shortening. Fundamental information is also needed on hydrogenation to permit production of improved modified fats and oils. Other areas in which markets for cottonseed oil need to be developed through research include fat emulsions for intravenous feeding and edible emulsifiers. Improved cocoa butter-like fats and other confectionery fats derived from cottonseed oil could also provide new markets for large quantities of oil, as could film-forming coatings for nuts, meats, and other food products to prevent moisture transfer. An additional urgent need is the preparation of cottonseed flours or protein concentrates for human consumption in developing countries to extend the supply of protein to meet the worldwide shortage. For further advances in utilizing cottonseed in food, information is needed in both the basic and applied areas of research.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving organic chemists, physical chemists, analytical chemists, biochemists, chemical engineers, and microbiologists engaged in both basic and applied studies on cottonseed and its products to develop new or extended food uses for these materials.

Research to develop fundamental information on the chemical composition and physical properties of cottonseed products is conducted at New Orleans, Louisiana, as a basis for efficient applied research to produce food products from cottonseed. Fundamental research on seed proteins and associated materials is being conducted by the Seed Protein Pioneering Research Laboratory. In other research, a major effort involves the isolation of cyclopropenoids in cottonseed and cottonseed oil, and investigation of the chemical and physical properties of these compounds with regard to their effect on the preparation and use of cottonseed products; the Foundation for Cotton Research and Education has contributed toward this research, and WU cooperates by testing the effect of the compounds in animals. Other in-house research involves development of new phase-relation and solubility data for unsaturated fatty esters and other fatty acid derivatives to obtain basic information needed to increase utilization of cottonseed oil. Additional research on chemical composition and physical properties of cottonseed and its products in relation to food is carried out under two contracts: (1) isolation and identification of the reaction products of gossypol with simple esters and model triglycerides, at Savannah State College, Savannah, Georgia; and (2) biological studies of cyclopropenoid derivatives and cottonseed oils treated to remove cyclopropenoids, at Ralston Purina Company, St. Louis, Missouri. Work in this area is also conducted under a grant to Boston University, Boston, Massachusetts, on the development of procedures for synthesizing C^{14} -labeled malvalic acid esters.

Research in the area of microbiology and toxicology as they apply to food products is being conducted at New Orleans, Louisiana. It concerns the identification, isolation, characterization, and analysis of mycotoxins elaborated by fungi indigenous to cottonseed.

Technology for the development of new and improved processes and products is the objective of research that is also conducted at New Orleans, Louisiana. In one current project, processes for preparing and characterizing cocoa butter-like fats and improving their performance are receiving attention. Another area concerns the development of new and improved techniques for preparing commercially valuable esters from cottonseed oil fatty acids. The Office of the Surgeon General is supporting research on fat emulsions for intravenous nutrition, which is conducted cooperatively with the U. S. Army Medical Research and Nutrition Laboratory and several medical school research groups. Other current work--supported by the Agency for International Development--involves a study of the preparation of cottonseed flours and their derived products for human consumption in developing countries. UNICEF cooperates by arranging nutritional evaluations of experimental products in developing countries, and the Human Nutrition Research Division, ARS, also cooperates by evaluating certain of the products. Informal cooperation is also maintained with industry in connection with the research on new and improved food products and processing technology.

Other research on chemical composition and physical properties is in progress under grants of P. L. 480 funds to the following foreign institutions: University of Rome, Rome, Italy, for basic investigations on the physical and physiochemical properties of cottonseed proteins (project duration - 5 years); Hebrew University, Hadassah Medical School, Jerusalem, Israel, for development of methods for the improved preparation of protein hydrolysates for the determination of amino acids, to provide a more accurate means for assessment of protein quality and nutritive value of oilseed proteins, thus contributing to their increased utilization (project duration - 3 years); Kyoto University, Kyoto, Japan; for an investigation of the chemical composition and reactivity of the nucleic acids of cottonseed, to obtain basic information needed for the increased utilization of this commodity (project duration - 3 years); Commonwealth Scientific and Industrial Research Organization, Ryde, Australia, for an investigation of the chemistry and biological effects of cyclopropenoid compounds that occur in cottonseed and its products (project duration - 5 years); and University of Bombay, Bombay, India, for a study of the synthesis and properties of pure saturated diacid and triacid triglycerides for use as model compounds in obtaining basic information needed to improve the utilization of cottonseed oil (project duration - 5 years).

Additional research in microbiology and toxicology is in progress under a grant of P. L. 480 funds to the following foreign institution: Nagoya University, Anjo, Aichi, Japan, for studies of the biochemical mode of action of aflatoxins and their biodegradation, to obtain basic information needed for control of these toxins in cottonseed and other agricultural commodities that may be exposed to contamination by Aspergillus flavus (project duration - 3 years).

The Federal in-house scientific effort devoted to research in this area totals 23.5 scientific man-years. Of this number, 12.8 are applied to chemical composition and physical properties, 2.6 to microbiology and toxicology, and 8.1 to technology--process and product development. The domestic contract and grant research involves an additional 2.3 scientific man-years, all of which are on chemical composition and physical properties. P. L. 480 research involves 6 grants, 5 of which are on chemical composition and physical properties, the remaining grant being on microbiology and toxicology.

The following lines of work were terminated during the year: (1) contract research on chemical transformations of olefinic compounds of fats and other agricultural materials by hydroboration and subsequent reactions to develop basic information for the production of useful products; and (2) a study of the relationship of substituent fatty acid groups on the physical properties of diacid triglycerides of palmitic and stearic acids as a means of increasing the utilization of cottonseed oil for food and industrial purposes (P.L. 480 project) (both under Chemical Composition and Physical Properties).

PROGRAM OF STATE EXPERIMENT STATIONS

A total of 0.3 scientific man-year is devoted to this area of research.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Chemical, Physical, and Biological Properties and Structural Factors of the Proteins. The composition, properties, structural factors, and reactions of oilseed proteins and associated materials are being investigated in a program of pioneering research conducted by the Seed Protein Pioneering Research Laboratory. The fundamental information developed should lead to new concepts and possibly new applications for oilseed proteins, including cottonseed proteins. Since peanuts were found to be an especially suitable experimental material and were employed for much of the early pioneering research on seed proteins, the report of progress in the research is given in Area 5, "Peanut Utilization - Food."

Under a P. L. 480 project now nearing completion at the Institute of Biological Chemistry of the University of Rome in Italy, a fundamental investigation of the physical and physicochemical properties of pure isolated cottonseed proteins has been conducted. In earlier stages of the work, two major proteins, designated Acaline A and Acaline B, together with a minor protein, Acaline C, were isolated from protein extracted from glandless cottonseed. Recent progress has been concerned with the further purification of the two major portions and with their characterization. Optical rotatory dispersion measurements have been obtained, and Acaline A has been subjected to tryptic digestion with a view toward the elucidation of its subunit structure. Curves illustrating spectropolarimetric analyses and a peptide map of Acaline A after chromatography and electrophoresis have been prepared. The fundamental information concerning the properties, subunit structure, and conformation of the isolated cottonseed proteins being obtained will have potential application in developing cottonseed proteins into foods suitable for human needs. (UR-E15-(40)-33).

Improved methods for the preparation of protein hydrolysates to permit the accurate determination of the amino acid makeup of cottonseed and other oilseed proteins are being investigated under a P. L. 480 project at the Hadassah Medical School of the Hebrew University in Jerusalem, Israel. Results obtained early in these investigations have shown that one hour's refluxing in 6N hydrochloric acid completely disperses the proteins in cottonseed meal and that the peptides in the hydrolysate are not precipitated by trichloroacetic acid. Work is underway to permit selection of enzymes for the complete hydrolysis of the peptides present in the hydrochloric acid extracts. Information of the type being sought is needed to permit accurate estimation of the amino acids present in various proteins. Present methods suffer from reaction of reducing sugars present with amino acids

containing alpha-amino groups, and the destruction or alteration of certain amino acids during hydrolysis. (UR-A10-(40)-52).

In a P. L. 480 project now in its initial stages at Kyoto University, Kyoto, Japan, the chemical composition and reactivity of the nucleic acids present in cottonseed are being investigated. The research is an outgrowth of, and employs methodology developed in, an earlier P. L. 480 project at the same institution concerned with the nucleic acids of sugarcane juice and sugar cane molasses. Sixteen nucleic acid components have been separated in mature cottonseed and have been identified. The presence of free nucleic acid components in mature cottonseed, not heretofore reported, has been demonstrated. Their presence has a real significance in both the physiology of the seed and the use of nitrogen content as a measure of protein content. Large amounts of phosphate, in excess of that accounted for by the base composition, have been found in the S-RNA fraction. The significance of this is being investigated. Basic information being developed will have practical importance in the storage of quiescent cottonseed, and in the analysis of cottonseed and its products. (UR-A11-(40)-29).

2. Chemical and Physical Properties of Cottonseed Pigments. By reacting gossypol with model methyl esters under conditions that made available the hydroxyl groups of the gossypol, scientists conducting contract research at Savannah State College, Savannah, Georgia, have shown that partial gossypol esters were produced, one of which was identified as gossypol hexastearate. These hydroxyl groups may therefore play a significant role in the color fixation reactions of gossypol with the triglycerides in the oil. (S4 1-138(C)).

3. Chemical, Physical, and Biochemical Properties of the Oil and Fatty Acids, Including Cyclopropene Fatty Acids. In cooperative work supported by the Foundation for Cotton Research and Education, further progress has been made toward preparing cyclopropenoid concentrates in amounts large enough for biological testing. A three-stage procedure has been developed for making 100-fold concentrates of the fraction of 1% cyclopropenoids in the methyl esters of cottonseed oil fatty acids. In pilot plant procedures, about 21 pounds of concentrate, averaging 5.2% cyclopropenoids, was prepared by solvent crystallization. Some of this concentrate was enriched to 9.6% cyclopropenoids by use of a York-Scheibel column, but at a loss of 79% of the cyclopropenoids. Also developed was an analytical method that for the first time permits the accurate determination of cyclopropenoids by a stoichiometric reaction rather than by reference to a standard. These techniques should facilitate the preparation of cyclopropenoid concentrates and the evaluation of their activity in biological tests. (S4 1-135).

In biological studies of cyclopropenoid derivatives conducted under contract by Ralston Purina Company, weanling rats and young laying hens were fed an experimental diet containing one of the following oils: refined and bleached cottonseed oil rendered Halphen-negative by a process developed at SU to

inactivate the cyclopropenoids by treatment with cottonseed fatty acids; slightly Halphen-positive cottonseed oil produced by conventional deodorization; the untreated cottonseed oil, which contained 0.52% cyclopropenoids; soybean oil; or soybean oil to which Sterculia foetida oil had been added. Preliminary results based on the hens' weight gain and egg production suggest that the special process for treating cottonseed oil is promising; however, continued evaluation is necessary. Also planned is a study of analytical methodology to quantitatively determine the presence of cyclopropenoids in animal tissues. (S4 1-137(C)).

In a grant to Boston University, initial research work has been focused on the development of suitable procedures for the synthesis of desired cyclopropenoid intermediates. Although slight modification of the best available synthetic procedures improved the yield of dialkylcyclopropenoids, such methods were not useful for the synthesis of esters of cyclopropenoid acids. Therefore, new synthetic routes were developed. At the present time, two esters of cyclopropenoid acids have been synthesized. These appear to be methyl 5,6-methano-5,6-hendecenoate and methyl malvalate, but further studies will be required to confirm these preliminary findings. If their use proves justified, the new procedures will comprise an important advance toward the ultimate synthesis of labeled isomeric malvalate esters. (S4 1-134(Gr)).

A P. L. 480 research project being conducted at the Division of Food Preservation, Commonwealth Scientific and Industrial Research Organization, Ryde, New South Wales, Australia, in which a study is being made of the chemistry and biological effects of cyclopropenoid fatty acids that occur in cottonseed and cottonseed products, is now entering its final stages. Means for incorporating C^{14} into cyclopropenoids were developed using seed slices, but the levels of activity achieved were not sufficiently high to be useful in feeding tests. Efforts to synthesize sterculic and malvalic acids labeled with C^{14} in the cyclopropene ring were not successful. Recent efforts have been devoted to the biological effects of cyclopropenoids and to the mechanism by which these inhibit the stearic acid dehydrogenase enzyme system. Malvalic and sterculic acids were shown to be effective at very low concentrations in causing irreversible inhibition of an enzyme system of chicken livers that desaturates stearic to oleic acid. Work is continuing to elucidate the inhibition mechanism, and to study the fractionation of the lipids from eggs of hens fed methyl sterculate. Information being obtained is urgently needed in evaluating the significance of the problem of cyclopropenoids in food and feed uses of cottonseed products. (UR-01-(40)-2).

Studies of the relationship of the substituent fatty acids to the physical properties of diacid triglycerides of saturated fatty acids that occur in cottonseed oil have been completed under a recently expired P. L. 480 grant at the University of Bombay in India. Members of the complete series of diacid triglycerides containing one or two molecules of palmitic or stearic acid and two or one of the even-carbon saturated fatty acids of the series from acetic to stearic acid, of both symmetrical and unsymmetrical

configuration, were prepared by appropriate methods of synthesis. Following their purification to about 99.8% purity, physical properties such as melting point of the most stable polymorphic form, density, molar volume, refractive index, molar refractivity, and viscosity were determined for each of the pure glycerides prepared. In addition, mixed melting points of these pure glycerides, taken two at a time, were determined. This extensive body of data concerning the physical properties of these highly pure triglycerides of known composition and configuration will provide a storehouse of fundamental information for use in the development of fats and oils whose composition and physical properties are tailored to special end uses in the area of food products and industrial materials. (UR-A7-(40)-3).

Research is just getting underway in a P. L. 480 project at the University of Bombay, Bombay, India, in which a study of the synthesis and properties of pure saturated diacid and triacid triglycerides for use as model compounds in obtaining information related to uses for cottonseed oil is being obtained. This research is an outgrowth of work under a previous P. L. 480 project, recently completed at the same institution, in which compounds of palmitic and stearic acids were studied, and is designed to cover the lower saturated fatty acids in the homologous series. Substantial progress has been made in the preparation and purification of the derivatives needed for the synthesis of the triglycerides, seventeen of which have now been prepared. Information developed concerning the properties of these model compounds will form the basis for further development of fats and oils tailored for special food and industrial applications. (UR-A7-(40)-124).

A novel graphical method has been developed for correlating and predicting solubility data for long-chain saturated and unsaturated fatty acid derivatives; complete solubility curves can now be constructed with only small amounts of material and a minimum of effort--two or three experimental determinations. This new "isopleth reference method" is even more useful than the previously reported isotherm and isopleth methods, since predictability is not limited to homologous compounds. Solubility data are of fundamental importance in the utilization of fats and oils, for they provide a method for selecting optimum solvents for the isolation and purification of long-chain saturated and unsaturated fatty acid derivatives by solvent crystallization or by liquid-liquid countercurrent procedures. They also afford a basis for choosing the best reaction media in physical and chemical modification procedures and in the development of industrial processes to produce valuable edible and inedible products from fats and oils. (S4 1-129, S4 1-143).

In an investigation of chemical transformations of olefinic compounds of fats, recently completed under contract by Purdue University, the selective hydroboration of 1-methoxy-3-hexene, followed by contrathermodynamic isomerization, oxidation, and hydrolysis, has been accomplished. This is the first step to a possible practical synthesis of omega-functional fatty acid derivatives, such as 18-hydroxy- or 18-aminostearic acid derivatives, which could provide good monomers for the manufacture of polyesters or polyamides. In addition,

the fundamental information obtained on amination via hydroboration opens a new field for the easy synthesis of stereospecific amines that could find uses in the pharmaceutical and pesticidal industries. (S4 1-112(C)).

B. Microbiology and Toxicology

1. Investigation of Fungi and Toxic Fungal Metabolites That May Occur in Cottonseed and Its Products. A fundamental investigation of the biochemical mode of action of aflatoxins and their biodegradation by plant cell systems is well underway in a P. L. 480 project at Nagoya University, Anjo, Aichi, Japan. Sliced sweetpotato tissue is being used as a substrate in the study of the biochemical mode of action of aflatoxins on plant cells, because the wounding of sweetpotatoes induces the production of enzymes. Aflatoxin B₁ was found to inhibit production of peroxidase and phenylalanine ammonia-lyase and to abnormally alter the production of *o*-diphenyl oxidase in the sweetpotato slices. It has been postulated that aflatoxin B₁ inhibits active operation of RNA on protein metabolism in response to wounding. Work under this project is expected to provide information that may be useful in the control of aflatoxin in cottonseed and other agricultural commodities. (UR-All-(40)-24).

Aflatoxins in cottonseed products can now be estimated by a new objective method that is more accurate and precise than the previous one. This recently developed method, which measures fluorescence of aflatoxins absorbed on thin layer chromatographic (TLC) plates, also features improved extraction, lead acetate-silica gel cleanup, and TLC development conditions. Partition chromatography on silica gel with methanol in benzene used in the immobile and mobile phases has yielded a superior technique for isolating aflatoxins B₁, B₂, G₁, and G₂. Studies initiated on the stability of aflatoxins in solution as judged by fluorescence and absorptivity showed marked differences attributable to solvent effects. Also, fluorescence solid state changes of aflatoxins B₁ and G₁ were more affected by ultraviolet radiation than were the B₂ and G₂ analogs. These data will be of extreme importance in preserving the identity and purity of primary standards and in providing more accurate and reliable analyses. In another phase of the research, hard seed coat cottonseed from the 1965 crop was shown to have little resistance to the invasion of *A. flavus* and elaboration of aflatoxin, whereas experimental hard coat seed grown in 1966 had a high degree of resistance during a 28-day incubation period. A study of strains of *A. flavus* that elaborate toxins showed that the relative proportions of B₁ and G₁ were significantly influenced by temperature, incubation time, and substrate. (S4 1-116).

C. Technology -- Process and Product Development

1. New and Improved Cottonseed Oil Products. Stearine from the solvent winterization of cottonseed oil was separated into nearly pure dipalmito unsaturated triglycerides and a fraction containing predominantly monopalmito diunsaturated glycerides. The dipalmito fraction was hydrogenated separately and further fractionated to yield cocoa butter-like fats. Procedures for

hydrogenating the monopalmito diunsaturated portion into a desirable component of cocoa butter-like fats are now being investigated. Because of the nature of the hydrogenation reaction, the separate hydrogenation and purification of these two fractions should produce a better cocoa butter-like fat than does similar processing of the whole stearine. Demonstration that a good cocoa butter-like fat can be prepared from cottonseed oil stearine has aroused considerable industrial interest, and a large processor of cottonseed has installed a pilot plant for evaluation of the SU method for producing cocoa butter-like fats. Since the process is relatively simple and since mixtures of these products with cocoa butter have the desirable property of melting completely when eaten, this development should provide a new market for a significant proportion of cottonseed oil. (S4 1-125, S4 1-142).

Research reported during the last period suggested that diglycerides of long-chain fatty acids, which are relatively difficult to prepare, could be prepared in a simple reaction of long-chain fatty acids, such as oleic, with the diglycerides of fatty acids whose chain lengths are as short or shorter than caproic, such as dibutyryl or diacetin. Almost pure dibutyryl was prepared with relative ease after it was found that the distribution coefficient of monobutyryl in the hexane-acetonitrile system was 0.0168 whereas that of dibutyryl was 0.2017. The purification was effected by a counter-current extraction employing five separatory funnels. In the esterification of dibutyryl with oleic acid, the reaction product contained 65% diglycerides, which analyzed 74% oleate, and 25% triglycerides, which analyzed 98% oleate. Reactions with diacetin are planned for the future. In a later phase of the research, all of which is designed to develop better processes for preparing commercially valuable esters from cottonseed oil, micro emulsions of sucrose were prepared. Although they are not yet stable under conditions of esterification, suitable modification--if successful--could lead to economical procedures for the preparation of esters of sucrose and other polyols. (S4 1-128, S4 1-128(Rev.)).

In research conducted cooperatively with the U. S. Army Medical Research and Nutrition Laboratory, as well as several medical schools, and supported by the Office of the Surgeon General, an emulsion of fractionated, pigment-free cottonseed oil stabilized with egg phosphatide has been prepared in amounts large enough for large-scale animal testing by cooperating institutions. Simultaneously, work is continuing to provide information on the stability of the emulsions after storage. Concentration of the autoxidation products of egg phosphatides was achieved to some extent by distribution between two solvent phases. The oxidation products tended to concentrate in a hydrocarbon phase and the unoxidized phosphatides in an aqueous ethanol phase. This result is important because autoxidation of egg phosphatides results in darkening, an increase in peroxide value, and development of a sharp odor. The conditions by which the creaming of emulsions can be precisely measured as rate functions, according to Stoke's equation, are being established. This will provide a comparative measure of the

physical stability of emulsions. (SU-O-O-4(3G)).

2. Cottonseed Flours and Derived Products for Human Consumption in Developing Countries. Progress has been made in research supported by the Agency for International Development (AID) and directed toward preparation of cottonseed flours and derived products for human consumption in developing countries. The causes of off-flavors in cottonseed flours prepared by extraction with solvent systems that contain acetone have been identified as diacetone alcohol and mesityl oxide, which are formed from the acetone. These components can now be detected in the solvents at levels as low as 1 ppm. Fractional distillation removes such impurities from commercial acetone. Furthermore, if cottonseed flakes are extracted at a relatively low temperature with an acetone-hexane-water mixture, very little diacetone alcohol and mesityl oxide are generated, as compared with processing at higher temperature. Thus flour with improved flavor can be prepared by modification of the acetone-hexane-water procedure or by use of another solvent, such as ethyl acetate. Use of liquid-solid cyclone separators to remove most of the pigment glands in solvent-damp hexane-extracted cottonseed flakes appears promising for producing a low-gossypol, high-protein flour in yields of 40% or more. (SU-O-O-3(AID)).

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^{1/} Publication resulting from research supported by funds transferred from the Office of the Surgeon General.

AREA 3 - COTTONSEED UTILIZATION - FEED

Problem. Cottonseed meal, used chiefly as a protein supplement in feeding ruminant animals, faces serious competition from synthetic urea and other supplements. The quality and nutritive value of the meal must be improved and new outlets developed to make optimum use of the meal produced from domestic cottonseed each year and to place it in a better competitive position with respect to the other protein feed supplements. The role of gossypol, a minor component of cottonseed, its interaction with various other components of cottonseed during processing, and its metabolic fate in monogastric animals need to be elucidated. Additional information is needed on the physiologically active constituents of the meal responsible for egg abnormalities, swine mortalities, and growth abnormalities of young animals that limit cottonseed meal's usefulness in poultry and swine rations, and for the reported implication of cottonseed meal in the incidence of trout hepatoma, which has resulted in its elimination from use in fish feeds in certain areas. Processing methods must be devised for the commercial production of meals that can be fed to broilers, laying hens, and swine, safely and without restriction.

A most important problem is the need for information on fungi and toxic fungal metabolites that may develop in cottonseed and its processed products. An urgent requirement is a practical method for inactivating or removing these toxic materials from contaminated seed, so that it can be utilized in feedstuffs rather than being diverted to use in fertilizers, which of course have a much lower value.

Much progress has been made toward the development of cottonseed meals that are fully suitable for all feeding needs. Additional basic and applied research is still needed, however, to develop practical and economic means of producing fully satisfactory meals during the processing of cottonseed without sacrificing the quality of the oil.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving organic chemists, physical chemists, analytical chemists, biochemists, microbiologists, and chemical engineers engaged in both basic and applied research to develop new or extended feed uses for cottonseed and its products.

Research to develop fundamental information on the chemical composition and physical properties of cottonseed products is conducted at New Orleans, Louisiana, as a foundation for efficient applied research pertaining to their use as feeds. Included in this area is fundamental research on seed proteins and associated materials, which is being conducted by the Seed Protein Pioneering Research Laboratory.

Certain areas of microbiology and toxicology as they pertain to cottonseed made into feeds are being investigated at New Orleans, Louisiana. One phase of this work is directed toward the inactivation or removal of aflatoxins from contaminated cottonseed and its products to permit their utilization in feeds. Another line of research is the isolation and identification of cottonseed constituents that cause mortalities among swine. Also, contract research is being conducted at Texas A&M University, College Station, Texas, on an investigation of the digestion and metabolism of gossypol by poultry and swine to obtain basic information needed to produce cottonseed meals fully suitable for use in rations for these animals.

Research on technology for developing new and improved processes and products is also being conducted at New Orleans, Louisiana. Processing conditions to produce high-quality oils and meals from glandless cottonseed are being investigated. Suitable processes and processing conditions are being developed to permit the inactivation of aflatoxin in cottonseed products by use of basic nitrogen compounds. In addition to the in-house work, contract research on technology is being conducted by IIT Research Institute, Chicago, Illinois, on development of practical processing methods for inactivation of cyclopropene groups, which decrease the value of cottonseed meal as a feed for laying hens.

Animal tests in connection with the overall research program are conducted through the cooperation of nutritionists in State Agricultural Experiment Stations at universities, in the Animal Husbandry Research Division, and in industry. The Pharmacology Laboratory at the Western Regional Research Laboratory, Albany, California, cooperates by conducting animal studies to determine the physiological and pharmacological effects of cyclopropene acids and toxic fungal metabolites. Cooperation is also maintained with the Crops Research Division, ARS, Market Quality Research Division, ARS, the Food and Drug Administration, the National Cottonseed Products Association, UNICEF, and members of the cottonseed industry.

In addition to domestic research, work on chemical composition and physical properties is conducted under a grant of P.L. 480 funds to the University of Madras, Madras, India; this is an investigation of the properties of the solvent system hexane-acetone-water to obtain fundamental information needed in the design of solvent recovery systems for use in an improved mixed solvent process for conversion of cottonseed to oil and meal (project duration - 5 years).

Other grants of P.L. 480 funds are being used for research on microbiology and toxicology by the following institutions: Instituto Farmacologico "Mario Negri," Milan, Italy, for a study of the mechanism of gossypol toxicity counteraction by L-lysine (project duration - 5 years); and Regional Cooperative for the Protection, the Development and the Practice of Fishing in Valle d'Aosta, Valle d'Aosta, Aosta, Italy, for experimental studies to elucidate the role of cottonseed meal in the induction of hepatoma in rainbow

trout to obtain fundamental information concerning the suitability of cottonseed meal for use in rations for this species (project duration - 3 years).

The Federal in-house scientific effort devoted to research in this area totals 11.1 scientific man-years. Of this number, 3.0 are devoted to chemical composition and physical properties, 4.1 to microbiology and toxicology, and 4.0 to technology--process and product development. The domestic contract research involves an additional 2.1 man-years, of which 0.8 are on microbiology and toxicology and 1.3 are on technology--process and product development. P. L. 480 research involves three grants, one on chemical composition and physical properties and two on microbiology and toxicology.

PROGRAM OF STATE EXPERIMENT STATIONS

A combined total of 2.6 scientific man-years is devoted to research on feed and industrial uses of cottonseed.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Fundamental Investigations of Cottonseed Proteins. The composition, properties, structural factors, and reactions of oilseed proteins and associated materials are being investigated in a program of basic research conducted by the Seed Protein Pioneering Research Laboratory. The information developed should lead to new concepts and possibly to new applications for oilseed proteins, including cottonseed protein. Since peanuts were found to be an especially suitable experimental material and were employed for much of the early pioneering research on seed proteins, the report of progress in this research is given in Area 5 - "Peanut Utilization - Food."

2. Basic Research to Improve the Nutritive Value of Cottonseed Meal. Protein concentrates have now been produced by dry, mechanical air classification of defatted flour from both glanded and glandless seed. A significant reduction in the gossypol content of glanded flour containing unruptured pigment glands can also be achieved by this process. Protein isolates prepared from glanded concentrates by calcium acetate extraction and dialysis were light in color and were more soluble and higher in protein than isolates produced from glandless flour by classical acid precipitation. Results with flours from glandless cottonseed are even more encouraging, for these concentrates can be directly introduced into the commercial market for use as foods. Grinding glandless flakes by turbomilling gave a greater reduction in particle size than Kolloplexing; however, since no significant concentration of any of the constituents measured was achieved by air classification, particle size per se cannot be used as a criterion in producing protein concentrates by this method. Other research indicates that the membrane

surrounding the protein body, not the solubility of the individual protein, is the determining factor in its extraction. The extent to which the selective permeability and solubility characteristics of the membrane are altered by processing the seed may account for the variability encountered when salt solubility and nitrogen solubility are used as predictive indices of the nutritive value of the protein of the seed. (S4 1-130).

B. Microbiology and Toxicology

1. Investigations of Undesirable Natural and Adventitious Materials That May Occur in Cottonseed Meal. Characteristics of the extraction of raw cottonseed flakes with the acetone-hexane-water azeotrope are being studied in relation to cottonseed constituents that cause mortalities in swine. The space distribution of oil in the raw cottonseed fragment exposed to the azeotrope has been described by a simple equation. This pattern appears to be the consequence of imbibition of the solvent and does not involve diffusion and surface phenomena. Since the movement of the oil is accomplished within ten seconds, the residence time of the marc in a countercurrent extraction need be no more than one to three minutes. Critical examination of the method for determining available lysine indicates that sampling errors may greatly exceed errors associated with experimental variables. It has also been determined that lysine may be destroyed in a reaction with nonreducing sugars, such as raffinose, which contain a 1,2-glycosidic linkage between glucose and fructose residues. This result may aid in preserving the lysine during the processing of oilseeds. Also, evidence that glucose amine remains in cottonseed meal extracted with hexane but is removed by acetone-hexane-water mixed solvent may explain the comparatively lower nutritional value of the hexane-extracted meal and thus may lead to improved processing of cottonseed. This research is conducted in cooperation with the Pharmacology Laboratory at WU and the Animal Husbandry Research Division, ARS. (S4 1-136).

Progress is being made in a P. L. 480 project at the "Mario Negri" Pharmacological Institute, Milan, Italy, on a study originally designed to determine the mechanism by which L-lysine counteracts the toxicity of gossypol fed to susceptible animals. Findings early in the research that L-lysine did not exert a protective action against gossypol focused attention on the need for other inactivating systems. Insight into the mechanism of toxicity of gossypol was gained by observation of the comparative effects of gossypol administration by intravenous injection, oral, and intraperitoneal routes. Both ferrous sulfate, as already known, and aluminum hydroxide were effective in protecting rats from acute toxicity and weight loss when fed gossypol, and in preventing intestinal hypertrophy. In addition, aluminum hydroxide prevented necrosis. This information has a direct bearing on current research efforts to alleviate the adverse effects of gossypol in the diets of susceptible animal species. (UR-E15-(40)-35).

In a P. L. 480 project at the Regional Board for the Protection, Development and Practice of Fishing in the Valley of Aosta, Aosta, Italy, studies

designed to determine the role of cottonseed meal in the induction of hepatoma in rainbow trout are being completed. Early results of the work confirmed the findings of other investigators that cottonseed meal per se in diets fed to rainbow trout does not cause the development of hepatomas. The causative factor was found to be aflatoxins in components of the diet, whatever the source. Long-term observations of trout fed diets containing aflatoxin-free cottonseed meal have turned up some slight specific lesions termed "cho-langitis." The possible role of cottonseed meal in the development of this disorder is now being investigated. The information developed in this project has already helped to clear cottonseed meal of implication in causing hepatoma when included in hatchery trout feeds. (UR-El5-(40)-44).

In other research on toxic components, extraction of aflatoxin-contaminated cottonseed meal with aqueous acetone appears promising for industrial application. Total aflatoxins were reduced from 180 ppb to 13 ppb in small-scale pilot-plant extractions with 90% acetone-10% water. No apparent processing difficulties were encountered with this solvent, but elevated temperature was required for efficient removal of aflatoxin. Sodium hydroxide and ozone were the most effective of the materials investigated for chemical inactivation, but these treatments require further evaluation. In another facet of the work, a method has been developed to permit use of a Coulter Counter for counting Tetrahymena pyriformis organisms in the presence of extraneous particles; this method will aid in measuring the growth response of the organism to protein-containing particles such as oilseed meals. (S4 1-133).

C. Technology--Process and Product Development

1. Processing Technology Directed Toward Improving Cottonseed Products.

In contract research on the inactivation of cyclopropenoids (CPA) conducted by the Illinois Institute of Technology, laying hens were fed cottonseed meals as prepared commercially or the same meals extracted with acetone-hexane-water to graded levels of residual CPA; their eggs have now been evaluated after three months' cold storage. Dietary levels as high as 5 ppm residual cyclopropenoids were tolerated with little, if any, adverse effect on egg quality as judged by yolk color, absence of pink whites, pH of yolks and whites, egg weight, and the stearic acid content and stearic:oleic acid ratios of yolk fats. This finding should foster increased use of cottonseed meal in poultry rations. Removal of residual cyclopropenoids from cottonseed meals by solvent extraction presently appears to be more efficient than chemical inactivation in preventing such abnormalities. (S4 1-117(C)).

In other research on the inactivation of toxic materials in oilseeds, cottonseed meal containing 210 ppb aflatoxins was treated with various amines; of those tested, methylamine and hydroxylamine were the most effective in lowering aflatoxins. Biological and nutritional tests, conducted at WU, should establish whether the treated meals can be used as feeds. (S4 1-139).

Bench-scale investigations of conditions of preparation before hexane extraction of meats from glandless cottonseed showed that very light-colored refined oils can be produced at preparation temperatures between 80° and 225° F. For glandless seed, unlike glanded seed, temperature had little effect upon the color of crude or refined oils. However, when these light refined oils were given a standard bleach, their color was not significantly lighter than that of oils from glanded cottonseed. Larger-scale studies are expected to confirm previous results with the oil and have already confirmed that a wide choice of temperature conditions in basket-extraction is available for producing meal or flour of highest quality as determined by chemical analyses. From these experiments, recommendations will be made for processing conditions in a mill-scale test. Results to date indicate that basket-extraction plants using direct hexane extraction can efficiently process glandless cottonseed without modifying their process or equipment. Because of the absence of gossypol, both the oil and meal products from glandless cottonseed should be of superior quality--the oil lighter in color and the meal of high quality for both feed and food uses. Thus glandless cottonseed offers an outstanding opportunity to the cottonseed industry to improve the quality and competitive position of its products and to expand and strengthen its markets. (S4 1-127).

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AREA 4 - COTTONSEED UTILIZATION - INDUSTRIAL PRODUCTS

Problem. To maintain and expand the consumption of cottonseed oil, it is necessary to extend areas of utilization. Increased markets can be developed through research and development work leading to the production of entirely new and valuable industrially useful products and chemicals from this oil. Research on industrial uses should be expanded to develop new large volume outlets, such as markets in coatings, plastics, plasticizers, lubricants, surface active agents, elastomers, pesticides, fungicides, and other chemical agents.

The preparation of synthetic organic chemicals consumes large quantities of raw materials and intermediates--billions of pounds per year--and represents one of the fastest growing chemical industries. Application of new reactions and techniques for chemically modifying fatty acids derived from cottonseed offers promise of expanding industrial markets. Research is needed to develop procedures for carrying out such reactions, to characterize the products obtained, and to evaluate their utility in a wide variety of industrial products.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving organic chemists, physical chemists, and analytical chemists engaged in both basic and applied research to develop new industrial uses for cottonseed and its products.

Research to develop new and improved industrial process and product technology is conducted at New Orleans, Louisiana. Present emphasis is on determination of the effect of the chemical structure of amide derivatives of long-chain fatty acids on their performance as plasticizers, lubricants, pesticides, and antifungal agents and in other industrial applications. Informal cooperation is maintained with industrial firms for the evaluation of promising research products for specific end uses.

Research on chemical composition, physical properties, and structure is carried out under grants of P. L. 480 funds to the following institutions: the Indian Institute of Science, Bangalore, India, for studies of the addition of carbenes to unsaturated fatty materials derived from cottonseed oil to provide possible new outlets for utilization of the oil (project duration 5 years) and for an investigation of chemical transformations of saturated fatty derivatives to alpha, beta-olefinic or tert-olefinic compounds, followed by hydroboration and suitable subsequent reactions, to produce useful products from cottonseed and new crop oils (project duration - 5 years); the Israel Institute of Technology, Haifa, Israel, for an investigation of π -complexed organometallic compounds derived from polyunsaturated fatty acids, to obtain fundamental information needed in expanding the utilization of cottonseed oil (project duration - 5 years); the Hebrew University Faculty of Science, Jerusalem, Israel, for an investigation of metalation reactions

for the modification of mono- and dienoic fatty acids to provide increased functionability, thereby leading to possible new industrial applications for cottonseed and other vegetable oils (project duration - 5 years); the Hebrew University of Jerusalem, Jerusalem, Israel, for a study of the preparation of new chemical derivatives from acrylonitrile and unsaturated fatty acids derived from cottonseed oil and other vegetable oils (project duration - 4 years); and the National Chemical Laboratory, Poona, India, for investigation of the synthesis and properties of new-type glycol mono alkyl ethers for control of water evaporation to extend the industrial utilization of cottonseed oil (project duration - 5 years).

The Federal in-house scientific effort devoted to research in this area totals 2.2 scientific man-years, all of which is applied to technology-- process and product development. P. L. 480 research involves 6 grants, all on chemical composition, physical properties, and structure.

PROGRAM OF STATE EXPERIMENT STATIONS

A combined total of 2.6 scientific man-years is devoted to research on industrial and feed uses of cottonseed.

PROGRESS - USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition, Physical Properties and Structure

1. Fundamental Investigations Directed toward the Development of Industrial Products. In P. L. 480 research at the Indian Institute of Science, Bangalore, India, a study of the addition of carbenes to unsaturated fatty acid derivatives of cottonseed oil is now nearing completion. Dibromocarbene addition reactions with methyl oleate have been carried out by various procedures, such as reactions with bromoform and anhydrous sodium hydroxide. The resulting addition products have been purified by inverted dry column chromatography, and have been identified by elemental analysis and various physical measurements. Similar reactions have been carried out with tert-butyl esters of oleic acid. Carboethoxycarbene addition to methyl oleate, cottonseed mixed methyl esters, and refined cottonseed oil have been conducted via ethyldiazoacetate reaction. Knowledge concerning the reactions by which the reactivity and chemical versatility of the fatty acids occurring in cottonseed oil can be increased should be useful in devising potential new industrial outlets for this commodity. (UR-A7-(40)-26).

Work is now nearing completion under a P. L. 480 project at the Israel Institute of Technology, Haifa, Israel, under which a study has been conducted of pi-complexed organometallic compounds derived from polyunsaturated fatty acids to gain information needed in developing industrial uses for cottonseed oil. Early progress in the investigation was concerned with the reactions of iron carbonyls with beta-ionone and analogous conjugated dienes and trienes that are precursors of Vitamin A, as model compounds. In more

recent stages of the work, experience with the model reactions has been applied to reactions of iron carbonyls in preparing pi-complexes of myrcene, alloocimene, and levopimaric acid. Reactions with beta eleostearic acid were found to yield two types of complexes, one of which retains a free olefinic bond in conjugation with the complexed diene system, the other in which the free double bond has moved out of conjugation. Final phases of the work may yield data that can be applied in producing industrially useful compounds from cottonseed oil derivatives. (UR-A10-(40)-34).

In a P. L. 480 project at the Hebrew University of Jerusalem, Israel, an investigation is directed toward metalation reactions with alkali and alkaline earth metals and their derivatives for modifying mono- and dienolic fatty acids from cottonseed oil and oils of selected "new crop" seeds to provide increased functionality. Attention in earlier stages of the work was centered on metalation reactions of ricinoleic acid, as a model compound. Recently progress has been made in the metalation-carbonation of linoleic acid derivatives which yielded a mixture of 9-, 11- and 13-carbomethoxy derivatives. Stearoyl methyl ether yielded 8-, 9-, 10-, and 11-carbomethoxy isomers and four dicarbomethoxy derivatives. These are being examined by mass spectrometry. Knowledge now being obtained should provide leads to cottonseed oil derived materials having potential industrial utility. (UR-A10-(40)-54).

Work is continuing in the final year of a P. L. 480 grant at the Hebrew University of Jerusalem, Israel, under which a study is being made of the preparation of new chemical derivatives from the reaction of acrylonitrile and fatty acids derived from cottonseed, tung, and several "new crop" oils. Studies during the earlier period of the grant were largely devoted to reactions of ricinoleic acid, as a model compound. Recently, attention has been directed to dienolic fatty acid esters of linoleic and alpha-eleostearic acids. Cyanoethylation of ethyl linoleate using Triton B and various alkali metal alkoxides as catalysts, as well as a weaker catalyst, pyridine, has been attempted without success. Attempts are planned using methyl or phenyl lithium as catalysts. Work is in progress to isolate fatty acids from Limnanthes douglasii oil supplied to the institution for further attempts at cyanoethylation. (UR-A10-(40)-53).

The synthesis and properties of newer-type glycol monoalkyl ethers based on cottonseed oil fatty acids for the suppression of water evaporation from the surface of storage areas is being investigated under a P. L. 480 project at the National Chemical Laboratory, Poona, India. Alkoxy ethanols and alkoxy propanols have been prepared from cetyl and stearyl alcohols obtained from fatty acids of cottonseed oil by hydrogenolysis. Evaporimeter studies have been conducted with surface films of these alkoxy alcohols and their mixtures. Physical properties such as surface viscosity, surface potential, specific resistance to evaporation, surface pressure area isotherms, equilibrium film pressure, and collapse pressure have been measured. Bulk state phase studies using differential thermal analysis, optical microscopy, and X-ray diffraction have been conducted. Evaporimeter studies are being extended

through the use of a wind tunnel with controlled wind velocity and temperatures to evaluate evaporation retardation under dynamic conditions. This basic information may lead to potential new industrial products from cottonseed oil for use in lowering evaporation losses from water reservoirs in dry areas of the U. S. (UR-A7-(40)-28).

B. Technology--Process and Product Development

1. Research to Develop New Reactions and Products Suitable for Industrial Use. Forty-nine N-mono and disubstituted amides have been prepared and characterized. Of the dialkylamides evaluated as polyvinyl chloride plasticizers, the N,N-dimethyl amides were highly efficient but were less desirable than the N,N-dibutyl amides because of lower compatibility tolerances, higher volatility loss, and poorer low-temperature performance. N,N-dibutyloleamide showed promise as a plasticizer-stabilizer for vinyl asbestos tile. In view of recent emphasis on broader areas of application, the other amides have been or will be screened as antimycotic and antifungal agents, as agricultural chemicals, or as fabric softeners, some of which may act as flame retardants. N-Oleoyl- and N-palmitoyl-N'-methylpiperazine appear to increase flex abrasion of resin-treated fabric and consequently are being further evaluated. Investigation of dihalogenation of the acyl moiety of N-substituted amides showed that it adversely affected thermal stability of amide plasticizers, but it did reduce volatility and extraction losses without affecting efficiency and low-temperature performance too severely. (S4 1-124, S4 1-140).

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AREA 5 - PEANUT UTILIZATION - FOOD

Problem. Peanuts constitute a major cash crop in the Southern States and are in surplus. Because of their high price, domestically produced peanuts are used primarily in foods such as peanut butter, confections, bakery goods, and roasted and salted nuts. A critical problem in the utilization of peanuts, which has recently been made more clearly evident, is the sporadic contamination of peanuts by toxin-producing strains of common molds. The possibility of toxins entering foods intended for human consumption, as well as feedstuffs, is of the utmost concern. Intensified research is therefore urgently needed on the isolation, identification, evaluation, control, and inactivation or removal of mold toxins, such as aflatoxin, that may develop in peanuts and processed peanut products.

New-type food products and improvement in the quality and uniformity of existing products are needed to increase consumer acceptance and extend markets for peanuts; the average per capita consumption has been rather stable since World War II. The increased trend toward mechanical harvesting has necessitated the use of artificial means for curing and drying peanuts, with the result that processed peanuts and peanut products do not always possess the same desirable flavor and physical properties as peanuts that have been cured slowly in the field. Information is needed on the physical and chemical characteristics of those chemical constituents in peanuts that affect flavor, aroma, and other important properties of the processed products, as a basis for developing new or improved products and processing procedures. Fundamental studies of peanut proteins and associated materials could similarly lead to the development of new concepts and new uses.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving organic chemists, biochemists, analytical chemists, microbiologists, and chemical engineers engaged in both basic and applied studies on peanuts and peanut products to increase consumer acceptance and extend markets for peanuts.

Research to develop basic information on the chemical composition and properties of peanuts, its constituents, and processed peanut products is carried out at New Orleans, Louisiana. As a part of the Seed Protein Pioneering Research Laboratory's research on various seed proteins, fundamental investigations of peanut proteins and associated materials are conducted to form the basis for developing new concepts and perhaps new uses for peanuts and peanut proteins.

Research on the flavor of peanuts and their processed products is also conducted at New Orleans, Louisiana. One objective is to separate and identify chemical compounds responsible for the characteristic aroma and flavor of freshly roasted peanut products, with special emphasis on the components that change in concentration as the roasted peanuts become stale. The Crops

Research Division of ARS, the Consumer and Marketing Service, and several State Experiment Stations cooperate in the research by providing samples of peanuts of known variety and history. Additional research on flavor is being carried out under contract at the Agricultural Experiment Station, Oklahoma State University, Stillwater, Oklahoma, on a study of the relation of the carbohydrate, amino acid, and protein components of the peanut to the formation of flavor and aroma during roasting.

Certain aspects of microbiology and toxicology as they relate to peanuts and their processed products are being investigated at New Orleans, Louisiana. An important line of such research is the identification, isolation, characterization, and analysis of mycotoxins elaborated by fungi that may develop in these products. Related research is concerned with the development of economically feasible methods for the inactivation or removal of aflatoxins from contaminated peanuts and peanut products to permit their utilization in foods (and feeds). Cooperation is maintained with the Crops Research Division, ARS, Market Quality Research Division, ARS, State Experiment Stations, the Pharmacology Laboratory of WU, the Food and Drug Administration, industry, and nutritionists in USDA, at universities, and elsewhere, in connection with this research. The problem of mycotoxins is also receiving attention in contract research at the Agricultural Experiment Station, Auburn University, Auburn, Alabama, on a study of the limiting environmental conditions for the elaboration of mycotoxins in peanuts; and at the Agricultural Experiment Station, Texas A&M University, College Station, Texas, to develop information relating to processing methods; preprocessing history; distribution of immature, mature, and germinating peanuts; and other conditions such as mold incidence as they affect consumer-use properties of processed peanut products.

Research on technology for the development of new and improved processes and products is being conducted at New Orleans, Louisiana. One project is concerned with the development of new and improved low-fat peanut products and processes for their manufacture. Informal cooperation is maintained with peanut suppliers and processors and with nutritionists and home economists for evaluation of experimental products as required. Other research, supported by the Agency for International Development, involves a study of the preparation of peanut flours and their derived products for human consumption in developing countries. Cooperation is maintained with UNICEF for arranging nutritional evaluations of experimental products in developing countries, and with the Human Nutrition Research Division, ARS, for evaluating certain of the products. Suitable processes and processing conditions for inactivating aflatoxins in peanut products by use of basic nitrogen compounds are also being studied; the necessary biologic evaluation is being done by the Pharmacology Laboratory at WU. Additional research on process and product development is being carried out under contract at the Agricultural Experiment Station, Oklahoma State University, Stillwater, Oklahoma, on a study of sterilizing or inactivating treatments in conjunction with artificial drying and curing of peanuts to develop processing conditions needed for producing mycotoxin-free roasted peanut products of optimum quality.

Other research on chemical composition and physical properties is in progress under a grant of P. L. 480 funds to the following foreign institution: The University of Granada, Granada, Spain, for an investigation of the rate of reaction of protein with carbohydrates in peanuts, to provide information leading to improved peanut products, thereby increasing the utilization of this commodity (project duration - 3 years).

The Federal in-house scientific research effort in this area totals 15.7 scientific man-years. Of this number, 2.6 are devoted to chemical composition and physical properties, 2.4 to flavor, 4.1 to microbiology and toxicology, 6.6 to technology--process and product development. The contract research involves an additional 2.3 scientific man-years, 1.0 on flavor, 0.7 on microbiology and toxicology, and 0.6 on technology--process and product development. P. L. 480 research involves one grant on chemical composition and physical properties.

The only line of work to be terminated during the year was contract research on the development of peanut products for use in preparation and fortification of processed and convenience foods (under Technology--Process and Product Development).

PROGRAM OF STATE EXPERIMENT STATIONS

A total of 8.8 scientific man-years is devoted to this area of research.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Chemical, Physical, and Biological Properties and Structural Factors of the Proteins. The Seed Protein Pioneering Research Laboratory is continuing its investigations of oilseeds, primarily peanuts. Research is currently centered on studies of ultrastructure, subcellular particles, new-found enzymes and proteins, immunochemical classification of proteins, and biochemical techniques.

The use of glutaraldehyde fixation and hexane extraction in addition to osmium and permanganate staining has expanded the amount of information obtained in studies of the ultrastructure of seeds with respect to the spherosomes, the oil-storage organelles of oilseeds. Osmium does not immobilize the membranes, but glutaraldehyde and permanganate do. Spherosomes from the peanut and the castor bean were isolated and examined by both techniques. Membranes stained with osmium only appear to swell, forming perfect spheres. By careful hexane extraction of the oil from fixed tissue, "ghosts" which show very distinct membranes were obtained. Storage of oil in nonhexane extracted spherosomes was confirmed by osmium staining.

Investigations of isolated and intact subcellular particles that contain the storage materials of oilseeds have been expanded to include cottonseed,

hempseed, and castor bean, in addition to those begun on peanuts some time ago. Information has been gathered on globoids of cottonseed and peanut, on the protein bodies (aleurone grains) of hempseed and cottonseed, and on spherosomes of peanut and castor bean.

Earlier work on globoids and protein bodies of cottonseed showed that the bulk of the phosphorous of protein bodies was localized in the globoids. This suggested globoids as possible storage cells of high-energy phosphate needed by the seed upon germination. Globoids of cottonseed contain about 3% protein with a glutamic acid/arginine ratio of 0.7. Peanut globoids are strikingly similar, having 3.7% protein with an acidic/basic ratio (aspartic and glutamic/lysine, histidine, and arginine) of 0.9.

Numerous improvements in techniques for isolation of intact protein bodies from seeds now permit these organelles to be purified readily. Protein bodies of cottonseed are 10% protein with a glutamic/arginine ratio of 1.5:1 (the reverse of that found in globoids). In peanuts, the protein bodies (11.6% N and 0.8% P) show the typical amino acid pattern of seed proteins: glycine/glutamic acid ratio of 0.04/1 and acidic/basic ratio of 2.2/1. Protein bodies from hempseed have been isolated and shown to contain pure edestin, the reserve protein, as crystalloids within these organelles--the first time a pure protein has been isolated from a protein body as a crystalloid. This edestin is composed of repeating polygonal-shaped units with a molecular weight of 300,000 (80 Å in size by electron microscopy).

Spherosomes were utilized in an attempt to correlate a biochemical reaction with in vivo morphological changes. By a combination of biochemical reactions and procedures with cytochemical techniques, spherosomes were isolated intact in the fatty layers after centrifuging homogenates of castor beans and peanuts. Lipase activity of the castor bean was localized with the spherosomes, suggesting this organelle as a possible seed counterpart of the animal lysosomes, which contain acid hydrolases. In contrast, peanut spherosomes were devoid of lipase activity, which was associated with the particulate fraction that sediments during centrifugation of the homogenate.

Evidence for several previously unreported enzymes in oilseeds has been obtained. For some, activity has been localized with known particles within the seed. Allantoicase, an enzyme heretofore found only in soybeans, has now been found in peanuts after controlled germination. This enzyme, which splits allantoic acid into glyoxalic acid and urea, was absent in the peanut until after five days of germination. Also, more sophisticated studies of protein bodies have revealed the presence of several enzymes localized there. Acid hydrolases are known to exist in animal lysosomes but were unreported in plants. During the past year, acid proteinase activity has been found associated with the protein bodies of hempseed, cottonseed, and peanut. This suggests that the occurrence of acid proteinase in quiescent seeds may be a universal feature. Another

acid hydrolase--acid phosphatase--was also found associated with the protein bodies of peanut and cottonseed. This enzyme is considered the principal "marker" enzyme of lysosomes. The identification of these two acid hydrolases associated with protein bodies is the first known isolation of lysosome-like particles from plant material with the so-called "latency" phenomenon.

The protein activator of castor bean lipase appears to be a glycoprotein. Its rather high acidic/basic amino acids ratio is similar to that found in castor bean allergens. By use of antisera for total proteins of the castor bean, the lipase activator and some of the characterized allergens were compared. These showed some similar precipitin bands and migration patterns. Specific antisera to the protein activator and to the total proteins are being prepared to determine if the activator is indeed one of the allergens. This would be the first known instance of a biochemical role of allergens in lipid metabolism.

Distribution and changes in the reserve proteins of the peanut before and after germination have also been studied by immunochemical techniques. Eight major and three minor antigenic constituents were detected in total extracts of dormant seed. Arachin, the reserve protein, is the main antigen. The α -conarachin fraction shows two antigenic components; one is anodic and the other is more cathodic than arachin. One antigenic constituent near arachin was characterized as catalase, and another, near α -conarachin, as amylase.

Proteins of the cotyledon and the axial tissue have also been compared by immunoelectrophoresis. Catalase, α_1 - and α_2 -conarachin, and the main constituents of arachin, are present in both tissues. Arachin concentration in the cotyledon, however, is four times that in the axial tissue. The most striking change noted was the absence in axial tissue of one constituent of the cotyledon migrating as α -conarachin. All other proteins detected appeared in both tissues.

By the upwards flow technique through a Sephadex column, the peanut globulin, arachin, has been separated into two well-defined fractions. The larger fraction is not homogenous, but modifications now underway may improve this. The smaller fraction is of nucleotide origin with a molecular weight of at least 300,000. In addition, a new procedure for isolation of almost pure arachin by low temperature crystallization has been developed. (SU P1).

Under a P.L. 480 project at the University of Granada in Spain, an investigation is being conducted of the rate of reaction of protein with carbohydrates in peanuts as influenced by moisture, oil content, time, and temperature during processing. Substantial progress has been made in the biological evaluation of the effect of roasting on the nutritive value of peanuts in feeding experiments with laboratory animals. Studies of the effect of roasting peanuts at various moisture levels indicate that

moisture may protect protein quality during roasting. These results suggest that maintaining high humidity during dry roasting of defatted peanuts might afford superior protein quality. Experiments have been suggested to determine whether sucrose, the nonreducing sugar normally present in peanuts, enhances the destructive action of heat on the nutritive value of the protein, such as is observed upon the addition of glucose, a reducing sugar, to peanut protein. Work done thus far with Spanish-type peanuts grown in Spain will be extended on a larger scale to the same type peanuts grown in the U.S. Eventually, the basic research being done under this project should lead to peanut products of enhanced flavor, aroma, and nutritive value. (UR-E25-(40)-19).

B. Flavor

1. Identification of Constituents and Factors Influencing Flavor and Aroma of Processed Products. In the investigation of lipid and lipid-soluble constituents of peanuts and their processed products, three phosphatide fractions and a triglyceride fraction obtained from peanut oil were purified adequately for analysis. The phosphatidyl ethanolamine fraction is strongly colored and usually rapidly develops a rancid odor. Thus this fraction may be of primary significance in the stability of oil and hence in its flavor. However, the development of a rancid odor in this fraction does not appear to be related to its fatty acid content, since analysis of the three phosphatide fractions and the triglyceride fraction showed that there are no marked differences in their fatty acid composition. Techniques developed in this research will be of value in separating the classes of lipids in peanut oil for evaluation of their individual contributions to the development of flavor during roasting and to flavor stability subsequent to processing. (S4 1-109(Rev.)).

The contractor (Agricultural Experiment Station, Oklahoma State University of Agriculture and Applied Science) has completed agronomic and organoleptic evaluation of flavor and aroma in the 1965 crop of shelled and unshelled Argentine variety peanuts cured under four conditions and stored under three conditions. Roasted nuts cured in windrows or at 90° F tasted better than those cured at 105° F or 120° F. Studies of chemical data on extracts of aleurone grains suggest that off-flavor bears a complex relationship to amino acids and to sugars. Agronomic data and data on curing and storing of the 1966 crop showed the same trends as previous crops. Data developed should permit selection of growing and processing conditions to yield products of maximum acceptability. The association of various compounds in the aleurone grains with the development of undesirable flavor and aroma upon roasting should provide an objective method for predicting the value of a given lot of peanuts. These data should also be of value to the plant geneticist in selecting the germ plasm for the breeding of peanuts having optimum roasting qualities. (S4 1-119(C)).

C. Microbiology and Toxicology

1. Investigation of Occurrence, Determination, and Properties of Fungi and Toxic Fungal Metabolites That May Develop in Peanuts and Their Processed Products. Contract research at the Agricultural Experiment Station at Auburn University has recently produced the finding that living peanuts, particularly when freshly dug, develop less aflatoxins than do sterilized nonliving peanuts when both types are inoculated with A. flavus and stored under the same conditions. The intact peanut shell affords considerable protection to invasion by mold and production of aflatoxin. Thus, culling peanuts with broken or damaged shells should decrease the possibility of contamination in peanut products. The limiting relative humidity of $86 \pm 1\%$ for aflatoxin production in cured peanuts on long term incubation at 30°C confirms previous results from short term experiments and suggests practical means for prevention of contamination. Other evidence obtained in this contract research indicates that varietal resistance may be involved in susceptibility to the production of aflatoxin. (S4 1-121(C)).

In-house research has demonstrated that when Spanish peanuts were inoculated with A. flavus and then stored for a year at 21°C and 65% relative humidity, there was essentially no change in the amount or relative proportions of the different aflatoxins. A study of the relation of the appearance of raw and roasted peanut kernels to aflatoxin content has been completed. In most cases the roasted half of each kernel that contained aflatoxin was darker than the roasted control, but color is not a good criterion for detecting the presence of aflatoxin. The observed decrease in aflatoxin upon roasting--apparent elimination when initial levels in the raw peanut were low--should offer reassurance to peanut processors and consumers. (S4 1-116).

In recently completed contract research conducted by the Agricultural Experiment Station, Texas A&M University, Spanish-type peanuts were studied with the ultimate objective of developing methods to process high-quality peanuts that are free of mycotoxins. The percentage of sound, mature kernels was lower in 1965 than in 1964, although the 1965 kernels were larger. In general, results from the 1965 crop confirmed findings determined for the 1964 crop: that roasted peanuts and peanut butter of superior flavor were obtained from peanuts grown on irrigated plots and that soil fumigation with pentachloronitrobenzene at planting had no deleterious effect on processed product quality and might actually improve flavor. The desirability of field curing or bag curing at ambient temperatures over oven drying at 120°F was also confirmed. Although none of the freshly harvested 1965 lots contained aflatoxin (over 50 ppb), after curing it was found in three bag-dried and one oven-dried lot. (S4 1-120(C)).

Total aflatoxins in a peanut meal (110 ppb) were reduced to none detectable in small scale pilot-plant extractions using 90% acetone-10% water solvent heated to about 120°F . Reductions up to 87% of the total aflatoxins have been achieved when a peanut meal assaying 256 ppb total aflatoxin was

extracted with 90% aqueous acetone in continuous pilot-plant equipment that simulated commercial extraction conditions and equipment. No apparent processing difficulties have been encountered using this solvent. A peanut processor is interested in carrying out a cooperative test to evaluate the process on a larger scale. Biological tests conducted at WU have confirmed that the 90% acetone-10% water solvent, as well as chemical treatments with sodium hydroxide and ozone, are effective in eliminating aflatoxins from contaminated peanut meal without drastic damage to the protein. Para-formaldehyde and dimethylol urea have also proved effective in reducing aflatoxins in the meal. (S4 1-133).

D. Technology--Process and Product Development

1. Peanut Flours and Derived Products for Human Consumption in Developing Countries. During the reporting period, cottonseed rather than peanut flours have received most of the attention in research supported by the Agency for International Development. However, samples of peanut flours have been shipped to various investigators who are cooperating in the evaluation of products designed for human consumption in developing countries. Plans include the development of practical processes to enable relatively small plants in developing countries to produce high-quality flours from both peanuts and cottonseed. (SU-O-O-3(AID)).

2. New Processed Products, Including Partially Defatted Peanut Products. Bench-scale investigations have been directed toward determining the effects of oil and air roasting variables on properties of partially defatted peanuts. To aid in this evaluation, new methods were developed for measuring true density as well as true expansion of the roasted peanuts. Another facet of the research showed that the loss of solubles in excess water during expansion of pressed peanuts increases with time of expansion. In cooperation with a commercial processor, cage presses were used to make partially defatted peanuts. Moisture content is the most important variable to control for maximum removal of oil, whereas age of the peanuts is apparently not a factor. Industrial interest is high--a number of companies are either commercially distributing partially defatted peanuts, in the process of developing partially defatted peanut products for commercial distribution, or producing and selling pressed peanuts for use in the new products. (S4 1-126).

The development of peanut products for use in processed and convenience foods has continued under contract to the Agricultural Experiment Station at Auburn University. Progress has been made in the preparation of screw-pressed peanut flours; evaluation of these flours in bakery, refrigerated, confectionery, and other products; new products in flake and extruded forms; and storage and shelf-life studies. The flours held up very well at room temperature and at 40° F, whereas meals and grits became rancid at room temperature after two to four months' storage time; oatmeal cookie mix became rancid after four months at room temperature but held up fairly well at 40° F. A peanut flake product appears to be potentially useful as

a high-protein breakfast cereal, and peanut products also may find use in such food items as muffins and snacks. A method was developed for preparing full-fat peanut flour, as well as flours with lesser amounts of oil. (S4 1-118(C)).

3. Methods Developed for Inactivating Aflatoxin or Removing It from Contaminated Peanut Kernels. The development of processing conditions to produce optimum-quality, mycotoxin-free roasted peanuts is the objective of a contract awarded to the Agricultural Experiment Station, Oklahoma State University. A reliable and precise laboratory procedure for screening fungicides for the inhibition of proliferation of A. flavus on peanuts has been developed. This technique shows that several commercially available fungicides (Phalatan, Difolatan, Duter, Captan, and Orthocide) appear effective on freshly harvested peanuts at a concentration of 100 ppm. However, the fungicides cannot be expected to be effective indefinitely. Work is now being conducted at fungicide concentrations of 12.5 ppm, and evaluations are being made on combinations of two or more inhibitors. Observations indicate that there is no close relationship between the rating of mold development and the concentration of aflatoxin. (S4 1-132(C)).

Suitable processes employing basic nitrogen compounds to inactivate aflatoxins are being investigated. Continuous ammoniation of prepressed solvent-extracted peanut meal containing 54 ppb aflatoxin B₁ and 18 ppb B₂, performed by a commercial plant in its Expander-Dryer, did not reduce the content of B₂; the maximum detoxification of B₁ was about 35%. However, treatment of the peanut meal with ammonia in a batch operation reduced both aflatoxins to trace quantities. Cooking peanut meal with 1.25% methylamine also appears to be promising. Biological tests conducted at WU have indicated that both ammonia and methylamine are effective in the detoxification of an aflatoxin-contaminated peanut meal, verifying the results obtained with thin-layer chromatographic assays for aflatoxins in the treated meals. Protein efficiency ratios were lowered by the treatments, but the effect was not drastic, and it appears feasible to utilize the detoxified meals in the usual channels, i.e., in the rations for farm animals. (S4 1-139).

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AREA 6 - CITRUS AND SUBTROPICAL FRUIT UTILIZATION - FOOD

Problem. The citrus and subtropical fruit production of the Southern

Region is an expanding industry with the need for the development of better--as well as new--consumer products and for the improvement of present or invention of new processing procedures and machinery. These advances are required to regularly utilize the currently large production, particularly of oranges and grapefruit, and the anticipated higher production of these fruits to the economic advantage of the growers, processors, and consumers. Basic research is needed to lay the groundwork for these advances. This research is needed, for example, on the composition and physical nature of essential oils; flavonoids, including bitter constituents; constituents responsible for oxidized off-flavors; carotenoids; and the like, which determine many of the sensory characteristics and affect product quality and stability. Other problems whose solutions are dependent upon the availability of more detailed compositional and physical data are: cloud stability, gelation, discoloration, and fermentation. There is also need of an adequate method for estimating the amount of peel solids in various citrus products to allow for better control.

Increased production of citrus has stimulated the development of new products, but many of these are urgently in need of improvement, which will depend in part upon advances in basic research. New products are needed to attract new markets and also to reduce packaging and shipping costs. Research is needed to improve frozen citrus concentrates as processing procedures change; to develop better high density concentrate products, citrus powders, chilled juice and section products, pulp-fortified products, and comminuted whole fruit products; and to develop new or improved canned products that have a natural fruit flavor. Research is especially needed on grapefruit to develop practical methods for reducing the bitterness and harshness of juice products and to increase the use of grapefruit juice base in mixed fruit juice blends, drinks, and concentrates. Along with progress on product development there is a serious need to improve the actual processing procedures, processing equipment, and packaging operations and materials to obtain and maintain the most desirable fruit characteristics, particularly for citrus powders.

In addition to the work on citrus, research is currently needed to develop new processed products from avocados.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving biochemists, organic chemists, and a chemical engineer engaged in both basic and applied utilization research studies on citrus and subtropical fruits of the Southern Region to develop new or extended uses for these commodities.

Research on chemical composition is conducted under contract by the Stanford Research Institute, South Pasadena, California, and is supervised by the

U. S. Food Crops Utilization Research Laboratory, Weslaco, Texas. It involves development of an oxalate or other organic acid index for estimating the amount of peel solids in orange and grapefruit paste, puree, and juice products.

Research to develop basic information on the chemistry of flavor of citrus and subtropical fruits and their products and byproducts is conducted at Weslaco, Texas, and at Winter Haven, Florida. This information provides the necessary basis for efficient research in developing new and improved food products and processing technology. At the Weslaco Laboratory, the program includes investigations of the influence of seasonal changes of carotenoid and flavonoid constituents that directly or indirectly affect flavor and color of processed products from Texas colored grapefruit, as a basis for improvement of processing characteristics of and products from these grapefruit. The Texas Agricultural Experiment Station (substation 15, Weslaco), Citrus Rootstock Investigations Laboratory (CR, ARS, Weslaco), the Texas College of Arts and Industries, and Rio Farms, Inc. (Edcouch) are providing grapefruit of known history and conducting--or cooperating in conducting--on-the-tree tests. At the Winter Haven Laboratory, the program includes: an evaluation of the flavor contribution of peel flavones to orange juice and identification of unknown peel flavones to develop improved orange juice products; investigations of the composition of essential oil in citrus products, particularly orange, to afford a basis for maintaining and improving the quality and uniformity of citrus products; a study of off-flavor development in processed citrus juice in relation to the lipid composition of the suspended matter; a study of the enzyme reactions in fresh and processed citrus to increase the natural flavor of citrus products; and exploration of means to minimize or block the formation of bitter components in grapefruit, a key step in developing processed grapefruit products of greater attractiveness to the consumer. Close consultation is maintained with the Florida Citrus Commission (Lakeland); the Florida Agricultural Experiment Station (Citrus Experiment Station, Lake Alfred); Citrus Research Investigations (CR, ARS, Orlando); Florida Citrus Mutual (Lakeland); and the citrus processing industry. Contract research on flavor, supervised by the Winter Haven Laboratory, is being carried out by the Ohio State University Research Foundation, Columbus, Ohio. It is a study of nonenzymic browning in model systems to develop methods of blocking the reactions involved in flavor deterioration of orange juice crystals.

Research to develop new and improved process and product technology is carried out at Weslaco, Texas, and Winter Haven, Florida. At Weslaco, comminuted whole fruit, powders, pastes, purees, and beverage products are being developed from oranges and grapefruit to improve processing efficiency and to diminish pollution problems. This Laboratory is also investigating factors that affect the stability of a freeze-dried avocado salad product, particularly the lipid constituents. The citrus and subtropical fruit research is being carried out in part in cooperation with several state and

private organizations. The cooperators provide fruit or raw materials, such as pulp and juice, of known history. Processing plant facilities are available from the Texsun Citrus Corporation (Weslaco) and Rio-Vac, Inc. (Harlingen). Formal agreements exist with the Texas Agricultural Station (College Station and Weslaco), with Texsun Citrus Corporation (Weslaco), and with Rio Farms, Inc. (Edcouch). Informal cooperation is maintained with Texas Citrus Mutual, Inc. (Weslaco), Texas Cannery Association (Weslaco), and other organizations as necessary for the procurement and processing of fruit. At Winter Haven, the application of freeze drying to citrus and other subtropical fruits to develop new and improved products is being studied. Also, improving the commercial utility of the foam-mat process for production of citrus crystals is being investigated. This latter research is conducted in cooperation with the Western Utilization Research and Development Division (ARS) and the Florida Citrus Commission under a formal memorandum of understanding. Contract research on process and product development, supervised by the Weslaco Laboratory, is being carried out at the Citrus Experiment Station, University of Florida, Lake Alfred, Florida. It pertains to the development of a practical and efficient pilot plant-scale process for the production of enzymatically debittered grapefruit juice and products with improved flavor, product stability, and storage characteristics.

The Federal in-house scientific effort at the Southern Division devoted to work in this area totals 17.8 scientific man-years. Of this total, 10.3 is devoted to research on flavor and 7.5 to technology--process and product development. The contract research involves an additional 0.6 scientific man-years on chemical composition and physical properties, 0.6 on flavor, and 0.3 on technology--process and product development.

PROGRAM OF STATE EXPERIMENT STATIONS

A total of 23.4 scientific man-years is devoted to citrus and subtropical fruit utilization research.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Flavor

1. Enzyme Reactions Involved in Development of Citrus Flavor. Enzyme studies directed toward increasing the flavor of citrus products were recently initiated. Several techniques are being investigated to isolate from grapefruit the mitochondria, which contain many of the enzymes--oxidoreductases--involved in the development of citrus flavor. Isolation and functional characterization of these enzymes should contribute to a practical method of improving the flavor of processed citrus. Orange and grapefruit juice vesicles are being assayed enzymatically for the pyridine nucleotides, which are coenzymes for most of the oxidoreductases and have other important functions in many biosynthetic pathways. (S3 2-60).

2. Chemical and Physical Properties of Flavoring Constituents of Citrus and Subtropical Fruit Products. In research on the composition of essential oils in citrus products, experiments have established the quantity of nootkatone that can be added to grapefruit juice to improve its flavor and aroma, a threshold of five or six ppm. However, if the level was increased much above eight ppm, an unpleasant taste resulted. In another area of the research, a number of constituents of cold-pressed orange oil have been isolated and identified. Two new ketones--piperitenone and 6-methyl-5-hepten-2-one--have been identified. Other oxygenates identified are carvone, nootkatone, α -sinensal, β -sinensal, decanal, undecanal, geranial, and neral. A new monoterpenoid aldehyde was also isolated, and a sesquiterpene fraction was identified as a mixture of valencene isomers. Anhydrous orange essence and terpeneless orange oil were prepared and the major components identified; these two materials are expected to improve the flavor of "foam-mat" dried orange juice. (S3 2-48, S3 2-55).

3. Investigation of the Bitter Constituents and Flavonoids in Citrus Products. Considerable amounts of the five known flavones from the neutral fraction of orange peel juice extract are needed for taste evaluation of their contribution to orange juice flavor. In addition to the heptamethoxyflavone and tangeretin already available, nobiletin has now been obtained in quantity. A method has been devised for preparing sinensetin and tetra-O-methylscutellarein, and efforts are continuing to obtain sufficient amounts for taste evaluation of their contributions to orange juice flavor. Fruit samples from orange trees of known variety and rootstocks are being selected, and their juice and peels frozen for eventual analysis of the bitter constituent limonin. (S3 2-47, S3 2-54).

Two ingenious ways to introduce radioactive precursors into grapefruit leaves and stems have been devised. One of the more promising methods includes a separation of the leaf from several centimeters of the main vein starting at the tip end of the leaf, leaving the leaf attached to the plant. The section of vein free of leaf is immersed in the solution of radioactive material, which is expected to be taken in through the vein. Another method consists in forming a hole in the stem in such a manner as not to cause air blockage of the phloem and xylem tubes, thus allowing for the possible uptake of a solution. The results of these experiments are not definitive as yet. This work will ultimately help determine not only the metabolic pathways of the synthesis of naringin, a bitter component, but also the subsequent use of possible inhibitors. In another phase of the research, the relation of age to the ratio of naringin to naringenin-7 β -rutinoside (tasteless) in leaves and fruit is being determined to provide information that could aid in the preferential conversion of naringin to its tasteless isomer. Tissue culture procedures and use of cell-free enzyme systems have greatly facilitated the study of the precursors, synthesis, and metabolism of naringin in grapefruit tissues. (S3 2-49).

Studies have continued on the influence of seasonal variations in color and flavor of Texas colored grapefruit on the quality of its processed products.

A new highly red-colored seedy grapefruit, Hudson Foster Pink, appears promising for yielding more colorful sections to be used in chilled and canned products--and over a longer harvesting time. Its frozen sections also undergo less weeping and softening than those prepared from Redblush grapefruit. Under extremely long storage, the Hudson does not become insipid as other red varieties do. The effect of fruit age and time of fruit set on flavonoid and liminoid content is also being studied. Preparation of a sample by hard-freezing it with dry ice, grinding both sample and ice, and then removing the ice may have commercial application for comminuting food products with resilient or tough tissue. In another area of the research, two methods have been developed for obtaining reasonably high and enduring flowrates through columns of Polyclar AT resin. One method, which produces a resin that absorbs flavonoids to a greater degree than the original resin, may therefore be more useful in debittering. (S3 2-51).

4. Development of Off-Flavor As Related to Lipid Composition. In preliminary research on a recently initiated project on off-flavor development in citrus juice as related to lipid composition, an off-flavor was noted in an orange concentrate stored for three months at 85°F, but there was no difference between the general lipid thin-layer chromatographic profile of this concentrate and that stored at 0°F. (S3 2-50(Rev.)).

B. Technology--Process and Product Development

1. Application of Foam-Mat Drying to Citrus. Cooperative research is being conducted with WU and the Florida Citrus Commission to improve the commercial utility of the foam-mat process for producing citrus crystals. Experimental foaming methods have made it possible to use very low viscosity concentrates that were previously unsatisfactory for foaming. Fourteen compounds isolated from the acid degradation of fructose have been identified as products of storage deterioration in orange crystals. Compounds formed from ascorbic acid may also be a source of off-flavor. Several types of flavoring additives, including essence, essence oil, and sweeteners, are compatible with foam-mat dried orange and grapefruit crystals. The storage life of orange crystals is lengthened slightly by use of higher oil levels or by the incorporation of grapefruit crystals. The fact that untrained tasters generally prefer higher levels of oil in orange crystals suggests a possible means of enhancing storage stability through the addition of oil. (S3 2-43, S3 2-59).

2. Process for Enzymatically Debittering Grapefruit Products. From contract research conducted by the Florida Agricultural Experiment Station is stemming the first evidence that an enzymatic debittering process has practical potential for reducing the bitterness of grapefruit products. Naringin in grapefruit juice can be reduced by immediate removal of the pulp, which represents only about 10% of the original volume but has a relatively high naringin level. Because of its small volume, the pulp could be economically debittered with naringinase. A feasible process may consist of removing the pulp from the juice, debittering it, and then adding it back. A low level

of naringinase could be used to debitter chilled grapefruit sections if they were stored for several weeks before use. (S3 2-46(C)).

3. Freeze-Dried Products from Citrus and Subtropical Fruits. Orange and grapefruit juices, strawberries, and guava paste have been freeze-dried in preliminary runs. There seems to be a direct relationship between the physical form of a product and the rate at which it may be freeze-dried. Melting point data were obtained on various food products by use of AC and DC resistance measurements; however, the difficulties inherent in these measurements for very low temperatures must be overcome before the data is reliable. Measurement of low temperatures was also attempted by differential scanning calorimetry, which gave more reliable values than those obtained by the resistance method. (S3 2-53).

Small samples of frozen avocado salad were freeze-dried to moisture contents varying from 0 to 8% and placed in storage at 100°F under an atmosphere of air. Analysis for peroxide values and free fatty acid contents after storage indicated that a product containing 2 to 3% moisture developed a minimum of peroxides and free fatty acids. The freeze-dried material reconstituted rapidly and retained its original fresh flavor. Also, semi-commercial quantities of frozen avocado salad were freeze-dried at a commercial plant and sealed in enameled cans under atmospheres of air, nitrogen, and vacuum. Air packs were judged unacceptable after the following storage periods: 100°F, 2 weeks; 70°F, 10 weeks; 40°F, 16 weeks. The nitrogen and vacuum packs stored at 70°F for 24 weeks were judged to have acceptable flavor. All samples stored at 100°F and 70°F were judged to have unacceptable flavor characteristics after 32 weeks' storage. (S3 2-52).

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AREA 7 - VEGETABLE UTILIZATION - FOOD

Problem. Although extensive progress has been made in recent years in developing stable, attractive, and convenient vegetable products, new and improved processed products must be developed and means of stabilizing perishable vegetables provided to minimize the adverse effects of seasonal surpluses and unfavorable markets and to provide an adequate supply of good food for a growing population. Product quality needs to be improved and processing costs reduced through the adaptation and application of the latest technological developments and nutritional findings. For example, a major problem of the cucumber industry, since most of the crop is brine-cured, is to improve the curing process so that no loss occurs in the value of the cucumber during brine curing and storage. Thus the cost of processing would also be reduced. New pure culture fermented products are needed to more fully utilize cucumbers and many other vegetables in attractive consumer items. Celery, already an important flavoring ingredient, could become much more important if the factors and constituents responsible for the intensity, variability, and stability of its flavor could be controlled in processing, and more flavorful and convenient products could be developed. A precooked, dehydrated, sweetpotato product that usually has good shelf life when sealed under an inert gas has been developed. It reconstitutes to a product having the characteristics of freshly cooked and pureed sweetpotatoes. However, applied research on a pilot-plant scale is needed to obtain additional engineering and processing data applicable to commercial production of flakes from sweetpotatoes of different variety and environmental history. Research is also needed to further improve quality and storageability of the product, particularly if packaged in air, and to provide the scientific basis for the development of a process for making excellent flakes from uncured, freshly dug sweetpotatoes. Another area that should be investigated is the possibility of developing new food products in which sweetpotatoes are combined with other foods.

To improve processed vegetables, there is a continuing need to investigate the characteristics of the raw material as they are affected by climate, soil, cultural practices, and breeding. Several vegetable crops, including sweetpotatoes, hot peppers, okra, and Southern peas, are grown almost exclusively in the Southern Region. Many other vegetables grown in warm climates differ in their chemical and physical characteristics from the same crops grown in the temperate regions. Tomatoes, for example, are frequently poorer in color, flavor, and texture. More utilization research is needed to complement the Federal and State production research programs and to provide cooperation in the form of composition and processing studies. This kind of cooperation is also needed to prevent the release of breeding selections that are entirely unsuited for processing.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving biochemists, organic chemists, microbiologists, food technologists, and chemical engineers engaged in both basic and applied utilization research studies on vegetables of the Southern Region to develop new or extended uses for these commodities.

Research to develop basic information on chemical composition and physical properties of vegetables, their products, and their byproducts is conducted as a basis for efficient research in developing new and improved food products and processing technology. Research conducted at and in cooperation with the North Carolina Agricultural Experiment Station, Raleigh, North Carolina, is concerned with basic investigations of the chemistry and biochemistry of the carotenoid pigments in vegetables in relation to variety, maturity, and environmental factors, to facilitate the development of improved and more attractive processed products. Additional research on chemical composition and physical properties is being carried out under a grant at the Research Triangle Institute, Durham, North Carolina, on elucidation of the molecular structure and chemical characteristics of the pectinase inhibitor that occurs in sericea forage and other plant sources and has proved effective in preventing softening of cucumbers in brine curing.

Investigations of factors influencing the flavor of fresh, acidified, and fermented vegetable products, with emphasis on cucumbers, are being carried out at the U. S. Food Fermentation Laboratory, Raleigh, North Carolina, to provide the basis for increasing consumer acceptability. The North Carolina and Michigan Agricultural Experiment Station and the Pickle Packers International, Inc., cooperate in this research.

In the development of technology for new and improved processes and products, both basic and applied research is being carried out at New Orleans, Louisiana. Research is being conducted on the use of sweetpotatoes in combination with other foods to produce new products, the incorporation of protein concentrates and other nutrients, and investigation of constituent changes produced by the enzyme α -amylase. Pilot-plant investigations are being conducted as a phase of this research. Cooperation is maintained with the Marketing Economic Division, ERS, for the market evaluation of improved products, and with the Louisiana Agricultural Experiment Station, the Louisiana Sweetpotato Association, the Louisiana Sweetpotato Commission, the North Carolina Yam Association, North Carolina State College, the Sweetpotato Council of the United States, and other associations and industry groups. Other research on process and product development is in progress at the U. S. Food Fermentation Laboratory, Raleigh, North Carolina. Current emphasis is on investigations of methods for the controlled fermentation of cucumbers and other vegetables by application of pure culture techniques to fermentation practices to reduce processing costs and improve product characteristics. Limited cooperative work is conducted to evaluate new

cucumber varieties (or selections) for processing into brine-cured and fresh-pack products. Cooperation is maintained with the North Carolina Agricultural Experiment Station. The Michigan State University (Department of Microbiology) is also cooperating by providing technical assistance in the controlled fermentation studies. The Pickle Packers International, Inc., also provides technical assistance and supplies raw material. The U. S. Food Crops Utilization Research Laboratory, Weslaco, Texas, is conducting research directed toward developing new and improved processed products from Southern-grown vegetables other than sweetpotatoes and celery. The Texas Agricultural Experiment Station, the Crops Research Division, ARS, and industry associations provide raw materials of known history for this research. In progress at the U. S. Fruit and Vegetable Products Laboratory, Winter Haven, Florida, is research on the development of an improved natural celery flavor from essential oil of fresh celery to expand and improve the use of this vegetable.

The Federal in-house scientific effort at the Southern Division devoted to research in this area totals 8.1 scientific man-years. Of this total, 2.2 is devoted to chemical composition and physical properties, 1.3 to flavor, and 4.6 to technology -- process and product development. The domestic grant research involves an additional 0.6 man-year, on chemical composition and physical properties.

PROGRAM OF STATE EXPERIMENT STATIONS

A total of 28.7 scientific man-years is devoted to this area of research.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Investigations of the Chemistry and Biochemistry of the Carotenoid Pigments in Vegetables. Research on the chemistry of carotenoid pigments in vegetables is continuing in cooperation with the North Carolina Agricultural Experiment Station. Geranylgeraniol--reported to be the final precursor for C₄₀ carotenes--has been synthesized for the first time in a -2-¹⁴C-labeled form. The availability of labeled geranylgeraniol and the results of its incorporation into plastids indicates that more efficient cell-free systems might be developed in which it may be possible to study carotenogenesis in greater detail. The pigmented carotenoids are of economic interest because all enhance the color of food processed from carrots, sweetpotatoes, colored grapefruit, peppers, and tomatoes; some have provitamin A activity; and some may be responsible for flavor deterioration in dehydrated products. Research on the latter characteristic has shown that in dehydrated high carotenoid food products off-flavor increases as carotene concentration decreases and thus may be caused by decomposition of products of the carotene. Chromatographically isolated fractions of a nonsaponifiable extract of Goldrush sweetpotatoes, presumed to contain precursors of off-flavor, were stored and then compared with the odor of good quality and

off-flavored sweet potato flakes. Of these fractions, the one associated with carotene epoxide was judged to be most similar to the off-flavored flakes. (S3 5-28).

2. Identification and Characterization of Inhibitor of Enzyme That Softens Cucumbers. In a grant to the Research Triangle Institute, research is being conducted on the pectinase inhibitor extracted from the leaves of sericea forage and previously identified as a polymeric pro-anthocyanidin based on delphinidin, which is produced by acid hydrolysis of sericin. This information is important in understanding its use in the control of enzymatic softening of brined cucumbers. Elemental analyses have not yet permitted a clear-cut empirical formula for sericin to be derived. However, additional information on the inhibitor structure is being obtained from its reactions with periodate, benzoquinone, sulfurous acid, and other compounds. Another recent achievement is the development of a dialysis purification procedure that is more efficient with respect to savings in time and recovery of active enzyme inhibitor constituents than was the countercurrent distribution previously used. (S3 5-24(Gr.)).

B. Flavor

1. Factors Affecting the Flavor and Aroma of Cucumbers and Fermented Cucumber Products. Basic studies conducted in cooperation with the Pickle Packers International, Inc., and the North Carolina and Michigan Agricultural Experiment Stations have led to evidence that process conditions of cucumbers prior to fermentation may affect the flavor of the fermented product. Direct evidence has been obtained to support the theory that the aldehyde largely responsible for fresh cucumber flavor is not present in appreciable amounts in whole cucumbers but is formed enzymatically upon cutting or crushing the fruit. Thus it may be possible to control the formation of the green, or fresh, cucumber flavor in pickle products by proper timing and handling of the slicing and blanching operations. In another phase of the research, head-space vapor analyses of fermented vegetables have been extended. The technique has revealed that the highly volatile components of pure culture fermented cucumbers and olives are qualitatively very similar. Monitoring the odor of gas chromatograph effluents and retention data for methyl sulfide indicates that this compound is of major significance in relation to the flavor of fermented whole cucumbers and green olives. (S3 5-29).

C. Technology--Process and Product Development

1. Investigations to Improve Quality and Lower Costs of Processed Cucumbers and Other Vegetable Products. The pure culture fermentation process developed for cucumbers and other vegetables has been successfully applied to green olives. Although additional developmental work is required for commercialization, the process offers promise of controlling serious spoilage losses in brined olives caused by gassy deterioration, butyric fermentation, and enzymatic softening. A dividend has also resulted from the study of pure

culture fermentation of olives: a single lactic acid culture of Lactobacillus plantarum that has far more longevity, salt tolerance, acid tolerance, and regeneration power than any obtained from pickle brines. It promises to be extremely useful for the inoculation of pure culture pickle fermentations. It has also been established that Manzanillo olives and four other varieties contain a bacteriostatic material that has a selective inhibitory action for lactic acid bacteria according to species, with L. plantarum being the most resistant of those tested. Discovery of this bacteriostatic substance could lead to a completely new approach to the control of fermentation processes in vegetables. This research is conducted in cooperation with the Pickle Packers International, Inc., and the Michigan and North Carolina Agricultural Experiment Stations. (S3 5-27).

Experiments conducted in cooperation with the Texas Agricultural Experiment Station to confirm and extend earlier observations from a previous project have shown that the application of calcium chloride at the early stages of processing, such as peeling, offers advantages in firming canned whole tomato products and in increasing the viscosity of canned tomato juice; this is an improvement over other procedures in use, which involve additives. However, a machine for peeling tomatoes in the solution should submerge them for a specifically selected period of time. Otherwise, the epidermal cells are cooked and the effectiveness of the firming treatment is reduced. This increased time in solution may account for the fact that the treatment was not as effective for the Chico variety as it previously had been for Chico Grande. (S3 5-30).

2. New and Improved Dehydrated Sweetpotato Products and Processes.

Knowledge of the characteristics of α -amylase and means for controlling its activity have made it possible to make uniform and acceptable flakes from Goldrush having varied histories, including freshly harvested sweetpotatoes, stored and freshly cured roots, and cured and stored roots. Activating the enzymes in uncured sweetpotatoes in piece form, rather than as a puree, produces a greatly improved flake product. Studies on the effect of processing variables and changes in the raw sweetpotato roots during storage have shown that α -amylase content, conversion temperature, and conversion time influence the sugar content of the flakes. The principal change during processing was the conversion of starch to maltose. Indications are that low solids concentrates of the α -amylolytic fraction in sweetpotatoes could be prepared in quantity, an achievement that may lead to a method for controlling and improving the characteristics of the flakes. (S3 5-31).

3. Development of Processed Celery Products of Improved Flavor and

Convenience. Swirling agitation was found to be the only method of successfully rehydrating conventionally dried celery and was also the best method for celery treated to promote rapid rehydration. The length of time required for good rehydration is still too long to be practicable for use in prepared consumer products. However, a distillation process for recovering oil from celery waste was developed, and future work will be confined to development of a better essential oil from fresh celery. (S3 5-23(Rev.)).

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AREA 8 - NAVAL STORES UTILIZATION - INDUSTRIAL PRODUCTS

Problem. More uses for pine gum, rosin and turpentine need to be developed through research to provide industrial markets for current and anticipated production of gum naval stores. The recent decline in use of gum rosin has resulted in the accumulation of a considerable surplus of this commodity. Other types of rosin as well as synthetic chemicals backed by strong industrial research programs have made serious inroads on the traditional markets for gum rosin. Gum turpentine is also faced with similar competition. If the turpentine farmers of the southeast are to continue to find profitable markets for their pine gum, existing knowledge of the properties of this commodity and its derived products must be used to develop new uses and strengthen old ones. New fundamental information about the chemistry of the terpenes and resin acids is also needed to fully exploit their unique characteristics. New or expanded uses for naval stores products are especially needed in polymers, plastics, elastomers, resins, plasticizers, surface coatings, textile finishes, odorants, insecticides, herbicides, and other large-volume industrial chemicals. There is also a serious need to improve existing processes and develop new processing technology for the industry.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program at Olustee, Florida, involving organic chemists and a chemical engineer engaged in both basic and applied research to discover and develop new and improved uses for pine gum and its products. In basic research on the chemical composition, physical properties, and structure of gum naval stores materials, emphasis is on the isolation and characterization of some of the unidentified components in pine oleoresin and gum rosin that contribute to the superiority of gum rosin over other rosins as an industrial material. In the area of process and product development, the research is directed toward the development of new and improved industrial products from pine gum, rosin, turpentine and their components, and suitable processes for producing these industrial products. Research is being conducted to develop economical processes for the preparation of polyglycols and polyethers from rosin, resin acids and pine gum for use in polyurethane elastomers and foams; to investigate the chemistry of the photoperoxides of pine gum to develop useful intermediates for the chemical industry; to explore various high-temperature reactions of gum rosin, rosin derivatives, resin acids and resin acid derivatives to develop new and unusual products having potential industrial utility; and to find practical methods for increasing the utility of terpenes and resin acids from pine gum by free radical addition of acids, aldehydes and other compounds to the olefinic bonds of these materials, thus increasing their functionality. Informal cooperation is maintained with other agencies, industrial firms, and universities in connection with the naval stores research program. The U. S. Forest Service cooperates by supplying selected samples of pine gum.

Additional research on process and product development is in progress under contract at the University of Florida, Gainesville, Florida, on the development of a practical process for the conversion of α -pinene to dimers in good yields, and the conversion of these dimers to useful, reactive derivatives; and at Battelle Memorial Institute, Columbus, Ohio, on investigations to expand the use of gum rosin and its derivatives in the adhesives industry.

Research in the field of chemical composition, physical properties and structure is in progress under a grant of P. L. 480 funds to the Juan de la Cierva School of Technical Investigations, Barcelona, Spain, for development of new or improved methods of preparing selected terpene alcohols for use as standards, to obtain basic information on the composition and properties of products made from pine gum (project duration - 3 yrs.).

The Federal in-house scientific research effort in this area totals 12.0 scientific man-years. Of this total, 0.2 is devoted to chemical composition, physical properties and structure and 11.8 to technology--process and product development. The contract research involves an additional 1.7 scientific man-years on process and product development. P. L. 480 research involves 1 grant for research in the field of chemical composition, physical properties and structure.

The following lines of work were terminated during the year: (1) The preparation of polymerizable monomers for vinyl and condensation-type polymers from terpene acids and terpene acid derivatives, and (2) Synthesis of terpene alcohols and glycols by reaction of formaldehyde with terpenes derived from gum turpentine, and study of use of these products in making polymers (both under Technology--Process and Product Development).

PROGRAM OF STATE EXPERIMENT STATIONS

One scientific man-year is devoted to this area of research.

PROGRESS--USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition, Physical Properties and Structure

1. Composition and Physical Properties of Pine Gum. Research to identify some of the unidentified components in pine oleoresin and rosin was recently initiated. Several samples of the neutral portions of oleoresin and rosin have been isolated and examined by gas chromatography. Preliminary indications are that these fractions are complex mixtures containing as many as 40 to 50 components. One of the major components has been isolated and tentatively identified as 3,5-dimethoxystilbene. (S5 2-60).

Under a P. L. 480 project now nearing completion at the Patronato "Juan de la Cierva" School of Technical Investigations, Barcelona, Spain, research has been conducted to develop new or improved methods for synthesizing, isolating

and purifying selected terpene alcohols for use as standards in instrumental methods for determining the composition and properties of products made from pine gum. Recent progress has resulted in the preparation of 7 previously unreported terpene 7- and 9-alcohols, in addition to some 30 such compounds reported earlier. These compounds, together with a number of purified intermediates, have been supplied to the Naval Stores Laboratory at Olustee, Florida, together with details of their synthesis, purification, analytical characteristics and spectral properties. These pure terpene alcohols of known structure will serve as standards, and provide much useful information for the instrumental analysis and characterization of naval stores products required in their processing into industrially useful materials. (UR-E25-(50)-36).

B. Technology--Process and Product Development

1. Development of Intermediates for the Production of Resins, Plastics, Plasticizers, and Other Industrial Products from Pine Gum and Its Components.

Research to produce useful chemicals from terpenes and resin acids by free radical addition of functional groups has continued. To provide a basis for selecting solvents and catalysts for free radical additions to resin acids, the stability at room temperature of levopimaric acid dissolved in various solvents, with and without added peroxides, has been investigated. No change in levopimaric acid was found when its cyclohexane solutions were stored for about 6 weeks; little change was observed for acetone solutions; but extensive isomerization occurred in carbon tetrachloride or methanol solutions. Isomerization in freshly distilled bromotrichloromethane was even faster than in carbon tetrachloride. Addition of benzoyl peroxide catalyst resulted in almost complete dehydrogenation of levopimaric acid in all solvents; di-t-butyl peroxide gave very little dehydrogenation, especially in cyclohexane and in acetone. Progress has been made in isolating and characterizing the products from the di-t-butyl peroxide catalyzed reaction of each of the following compounds with diethyl phosphite: limonene, carvomenthene, camphene, α - and β -pinenes, myrcene, abietic acid and levopimaric acid. Derivatives of this type may prove useful as stabilizers and plasticizers, fire retardants, and oxidation inhibitors. A copper-alkali system recently discovered in other research may provide a lower cost alternate to peroxides as the catalyst for addition of carbon tetrachloride to terpenes. (S5 2-57).

Good progress has been made in the research to convert rosin, resin acids, and pine gum derivatives into polyols for use in polyurethane applications. Hydrogenation has proved particularly useful for stabilizing formaldehyde-modified rosin from loss of formaldehyde and should enable the facile preparation of useful polyols from rosin. Hydrogenation and subsequent saponification of the acetate resulting from the reaction of abietic acid and paraformaldehyde gave a stable mixture containing about 60% of dimethylolated and 35% of monomethylolated products and 5% of unreacted acid. Reduction of the carboxyl function produced a mixture of a di- and triol. The reaction of propylene oxide with various hydroxymethylated

rosins and with the acrylic, maleic and fumaric adducts of levopimaric acid and of rosin has resulted in a large number of hydroxylated materials, some of which should have value as adhesives, polymers, and polymer modifiers. Vinyl esters of various hydrogenated rosins are being studied to determine which rosins will be most useful in the polymer field. (S5 2-56).

In contract research (University of Florida) optimum conditions were determined for dimerization of α -pinene with phosphoric acid catalysts. . It was demonstrated that this dimer has a highly hindered tricyclic structure with one tetra- and one trisubstituted double bond. The diepoxide prepared from the dimer has an unusual structure and may have potential in polymer applications. A second dimer from the phosphoric acid catalyzed dimerization is being identified. The structures of the dimers prepared with boron trifluoride-phenyl dichlorophosphine as the catalyst were also established. Indications are that these dimers should be more reactive and possibly more useful as chemical intermediates. They may have utility as ultraviolet screens or stabilizers. Titanium tetrachloride-nitromethane gave good yields of dimer and the product had only two major components. The cost of catalyst in this system is also favorable. Zinc chloride-nitromethane was found to be a particularly attractive and effective catalyst for the dimerization of α -pinene. This catalyst is cheaper and more convenient to use than titanium chloride-nitromethane, and it produces a simpler dimer mixture with less isomerization. Products obtained by dehydrogenating and oxidizing the dimers are being characterized. (S5 2-49(C)).

2. Conversion of Rosin Acids, Pine Gum and Turpentine into New Polymers, Protective Coatings, Resins and Plastics. In recently initiated research, high-temperature rearrangements or pyrolysis of gum rosin, resin acids, and their derivatives are being investigated as a basis for producing new materials of potential industrial utility. Of several approaches investigated for high-temperature rearrangements of rosin and resin acids, the passage of these materials through a hot tube at 1000°C offers the most promise and is currently receiving further study. It has been found that on heating abietic acid, gum rosin, wood rosin, or tall oil rosin above 180°C, an equilibrium mixture of abietic, palustic and neoabietic acids is rapidly formed. Because of the formation of this equilibrium mixture, the pyrolysis at 1000°C of any one of the four pure conjugated dienic resin acids may give the same results. (S5 2-58).

In continued research on epoxides and ozonization products of resin acids and their derivatives, a large number of compounds were prepared for testing as agricultural chemicals at the University of Florida. Hydrogenation of maleopimaric acid has been explored as a means of producing improved heat-resistant polyimide-amides from this acid. A large manufacturer of heat-resistant plastics is strongly interested in the use of polyimide-amides from maleopimaric acid as a modifier for one of their existing commercial products. (S5 2-52).

Mono-esters prepared by condensation of ethylene oxide or propylene oxide with rosin have been found to have a high abietyl content and to react readily with dienophiles. Because of their high reactivity and lower cost than esters prepared by direct esterification of rosin and glycols, these mono-esters should be attractive intermediates for preparing high molecular weight polyester resins. A promising new type of polyester has been obtained by reacting rosin with permaleic acid to produce rosin epoxides followed by further esterification of the maleic acid formed. A ready source of pimaric acid has been provided by the discovery of a new method for isolating this acid, as its 2-aminoethanol salt, from pine gum. (S5 2-53).

Research investigations to expand the use of gum rosin and its derivatives in the adhesives industry are being initiated under contract at Battelle Memorial Institute. Initially, four selected rosin-based products, including ethylene-gum rosin copolymer and hydrogenated rosin-ethylene copolymer, will be evaluated as components of four specific types of adhesive classes. Other products to be tested are being prepared. (S5 2-59(C)).

A rapid, convenient process for the preparation of levopimaric acid transannular peroxide has been developed. It utilizes singlet oxygen generated by the reaction of hydrogen peroxide and sodium hypochlorite. Unfortunately, use of the technique on pine gum and gum rosin gives products which contain low amounts of peroxide in comparison with that achieved by photosensitized oxidation. Several additional reactions of levopimaric acid transannular peroxide, including those with cyclohexylamine, α -pinene, mineral acids and carboxylic acids, have been explored. (S5 2-61).

In contract research at Cornell University, good yields of primary alcohols have been obtained by reacting formaldehyde with camphene, limonene, and α -terpinene. The processes for making these terpene alcohols are simple and practical, and the products are attractive chemical intermediates. It has been demonstrated that they can be converted to high-boiling diesters and polymerizable methacrylates. (S5 2-46(C)).

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AREA 9 - SWEET SORGHUM UTILIZATION - FOOD

Problem. The Lower Rio Grande Valley, which is largely dependent on an agricultural economy, must have a greater selection of crops for diversification to meet unfavorable environmental and marketing conditions that frequently beset the area. Freezes and hurricanes have destroyed many citrus groves and generally retarded citrus production--a valuable source of farm income. Cotton, a mainstay crop, is also a surplus crop. In addition, yields in the Valley are frequently low, and insects and root rot pose troublesome problems. Many vegetables do well, but heavy losses have resulted from freezes, heavy rains, and maturation times that place Valley vegetables in direct market competition with those grown in other parts of the U. S.

Sweet sorghum has potential for becoming a profitable diversification crop. There are now available new disease-resistant varieties with high sugar content. This factor, together with favorable world sugar prices, has encouraged consideration of sweet sorghum canes as a potential sugar crop for the Valley. The modest water requirements of sorghum and the subtropical climatic conditions conducive to an extended growing season increase its attractiveness. In addition, preliminary studies in processing encourage evaluation of this crop for the production of sugar; its process integration with those for beet and sugarcane would extend the use of costly raw sugar installations.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving an organic chemist engaged at the U. S. Food Crops Utilization Research Laboratory, Weslaco, Texas, in investigations of methods for purifying sweet sorghum juices to permit practical recovery of their sugar content. Close cooperation is maintained with Substation 15, Texas Agricultural Experiment Station, Weslaco, Texas, in growing and harvesting the breeding stock provided by the Crops Research Division.

The Federal scientific effort at the Southern Division devoted to research in this area totals 1.3 scientific man-years. All of this effort is on chemical composition and physical properties.

PROGRAM OF STATE EXPERIMENT STATIONS

A total of 6.0 scientific man-years is devoted to sweet sorghum and sugar crops utilization research.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Investigations of Chemical Characteristics of Sweet Sorghum to Evaluate Its Potential for Recovery of Sugar. Analysis of sweet sorghum field samples from the 1966 harvest are nearly complete. Because of their respective 1966 climates, sugar yields per ton of stalks were superior in the San Antonio-Winter Garden area but low in Weslaco; conversely, stalk tonnages per acre were low for the former but high for the latter, so that the values of sugar per acre were similar for the two areas. Modification of an analytical procedure for measuring the juice free acidity of field samples has permitted estimation of the clarification performance of such juices. One lot of sweet sorghum juice provided by the diffusion of finely chopped low sucrose silage-type stalks (with leaves) in the factory facilities of a sugar beet company was successfully clarified by the procedure proposed as a result of previous research. Data derived in this research have indicated approaches of significant value both in the selection of breeding lines and horticultural practices and in establishing suitable processing techniques. This information is being developed in cooperation with the Texas Agricultural Experiment Station at Weslaco and the Crops Research Division. (S5 5-51, S5 5-55).

PUBLICATIONS -- USDA AND COOPERATIVE PROGRAMS

None.

AREA 10 - RICE UTILIZATION - FOOD

Problem. The productive capacity of U. S. rice growers has increased faster than domestic and export consumption over the past decade, thus limiting the income potentially available from this major world food grain. Detailed knowledge of chemical composition and physical properties as related to processing is needed to guide milling, processing, and product development of U. S. rices so that they can better meet the quality and new product requirements needed for expanded markets. New and diverse food products that are economical to manufacture, convenient to prepare, and attractive in flavor and texture are needed to increase the total consumption of rice both domestically and abroad. Additional needs include the development of improved milling machinery and techniques, primarily to increase the yield of head rice; intensified research on deep milling to evaluate and utilize the protein flour and residual kernels produced by this technique; and research to provide greater flexibility in the industry by developing from either medium or long-grain rice new products that can be cooked to provide discrete kernels or as a gelatinous food.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program at New Orleans, Louisiana, involving biochemists and analytical chemists engaged in research on the chemical composition and physical properties of rice and its products. Research is being conducted to improve the potential of rice for new food, feed, and industrial products in relation to the physical properties and chemical composition of whole and fractionated kernels. Close cooperation is maintained, under formal memoranda of understanding, with the Louisiana, Arkansas, and Texas Rice Experiment Stations, who supply rice samples of known variety and cultural history for the experimental studies. The Rice Inspection Service, Grain Division, C&MS, New Orleans, Louisiana, cooperates by providing assistance in grading rice samples from the research investigations. Cooperation is also maintained with the Western Division.

Other research on chemical composition and physical properties is also being conducted under a P. L. 480 grant to Kyoto University, Kyoto, Japan. Scientists at this institution will study the distribution of the major proteins of rice within subcellular particles and the distribution of these particles in the cellular structure of the rice kernel to obtain basic information needed to develop new and improved rice products and methods of producing them (project duration--3 yrs.).

The Federal in-house scientific effort devoted to research in this area totals 2.0 scientific man-years, all of which are presently applied to chemical composition and physical properties.

Under P. L. 480 research there is presently one grant, also on chemical composition and physical properties.

PROGRAM OF STATE EXPERIMENT STATIONS

A total of 0.8 scientific man-year is devoted to this area of research.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Investigation of the Distribution of the Proteins and Other Constituents in the Rice Kernel. Under a P. L. 480 project at Kyoto University, Kyoto, Japan, a study is underway on the distribution of the major proteins of rice within the subcellular particles, and the distribution of these particles in the cellular structure of the rice kernel. Proteinaceous subcellular particles 1 to 4 microns in diameter, of oval or spherical shape, have been revealed by electron microscopic examination of rice endosperm. These particles have been isolated in fairly pure state and in good yield by differential centrifugation in density gradient media, after mechanical or enzymatic disintegration of the endosperm cells. The isolated particles are about 60% protein, dry basis, the remainder being lipids and carbohydrates. The investigators have postulated that these particles are the protein reserve subcellular particles in the rice endosperm. Research is now underway to develop better methods for the characterization of major proteins and to compare the protein fractions of white rice with those in rice polish. Basic information gained in the investigation will be of use in developing high-protein fractions from rice for use as enriching components of foods. (UR-A11-(10)-23).

Research has continued on the chemical composition and physical properties of high-protein rice flours prepared in a commercial Satake mill, a Japanese machine designed for the deep milling of rice in the manufacture of sake. Determination of levels of mineral nutrients showed that phosphorus, sulfur, potassium, calcium, and iron were considerably greater in the flours than in the original rice and residual kernels, whereas the level of copper was lower in the flours. Rice deep milled to about 6-7% weight removal had improved cooking quality. Machine variables of the Satake mill are being studied to determine optimum and economic conditions of operation with long- and medium-grain rices for the production of high-protein flours. In another phase of the research, an expanded, flavored rice snack has been prepared; it is similar to corn snacks but has a more desirable uniform tender texture. Also, a gelatinized rice flour that is soluble in cold water and is suitable as a thickening agent in mixtures with oilseed flours as a beverage or gruel appears particularly attractive for rice-eating developing countries. This research is conducted in cooperation with the Louisiana Agricultural Experiment Station. (S1 4-13, S1 4-14).

PUBLICATIONS -- USDA AND COOPERATIVE PROGRAMS

Chemical Composition and Physical Properties

Normand, F. L., Soignet, D. M., Hogan, J. T., and Deobald, H. J. 1966.
Content of certain nutrients and amino acids pattern in high-protein rice
flour. Rice J. 69(9), pp. 13-18.

General

- Anon. 1967. Proceedings of the national rice utilization conference,
April 5 and 6, 1966. U. S. Dept. Agr. ARS 72-53, 80 pp.
- Fisher, C. H., Kopacz, B. M., and Decossas, K. M. 1967. Research
capabilities at the Southern Utilization Research and Development Division.
Rice J. 70(5), pp. 28-35.
- Hogan, Joseph T. 1965. Rice research at the Southern and Western
Utilization Research and Development Divisions, U. S. Department of
Agriculture. J. Food Sci. Technol. (India) 2, pp. 98-102.
- Hogan, Joseph T. 1966. Rice utilization research at Southern Laboratory.
Rice J. Ann. 69(7), pp. 65-67.
- Hogan, Joseph T. 1967. Utilization of by-products of the rice milling
process. Rice bran, oil and wax. In "Rice By-Product Utilization,"
Food Agr. Organ. U.N., FAO, Land Water Develop. Div., Informal Working
Bull. 30, pp. 1-6.

AREA 11 - DECIDUOUS FRUIT AND TREE NUT UTILIZATION - FOOD

Problem. The peach industry in the Southeastern United States is dependent to a large extent on the fresh market. A peach processing industry is needed in the Southeastern states to provide a profitable market for more of the edible peaches that do not meet fresh market standards and to rapidly convert a higher proportion of the overall crop to stable forms. Additional basic information on the flavor components of peaches is needed to guide development of improved processed products from southern grown fruit.

Climatic conditions that cause rapid deterioration of fresh peaches both on and off the tree, erratic ripening periods and markets, and short-lived peach orchards are other factors contributing to the need for more extensively integrated fresh market-processing operations. Technical problems preventing the more rapid development of the peach processing industry in the Southeastern states must be overcome. Many of the peach varieties grown in the Southeast require a modification of processing procedures to make satisfactory standard products. Still other varieties cannot be used in these products, and new food forms must be found for them. Recent rapid advances in food science and processing technology make it possible through research to develop both new and improved peach products. These are needed to bolster the economics of the South's peach industry, as well as to provide the superior qualities and greater convenience that the consumer now demands in food products.

USDA AND COOPERATIVE PROGRAM

The research effort in this area has been terminated. It had been conducted under one contract designed to develop basic information on the flavor of peaches, particularly varieties grown in the Southeastern states (under Flavor).

PROGRAM OF STATE EXPERIMENT STATIONS

A total of 38.5 scientific man-years is devoted to this area of research.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Flavor

1. Basic Studies on Flavor and Aroma Constituents of Peaches. The Georgia Agricultural Experiment Station has completed its contract research on flavor and aroma constituents of peaches. Gas chromatography of acids isolated from three varieties of peaches at three stages of ripeness confirmed observations that malic and citric acids were major components, the latter predominant in

the shipping ripe stage, the former at the soft ripe stage. Nine ester components of peach flavor were identified by gas chromatography and infrared spectroscopy. (S3 2-44(C)).

PUBLICATIONS -- USDA AND COOPERATIVE PROGRAMS

Technology--Process and Product Development

Wadsworth, J. I., and Spadaro, J. J. (SU); Boggess, T. S., Jr., and Heaton, E. K. (Ga. Agr. Expt. Sta., Experiment, Ga.). 1966. Flaking process could protect profits on rejected peaches. Canner/Packer 135(10), pp. 28-29.

Line Project Check List - Reporting Year July 1, 1966 to June 30, 1967

| Work & Line Project Number | Work and Line Project Titles | Work Locations During Past Year | Line Proj. Incl. in | |
|----------------------------|--|---------------------------------|---------------------|-------------------|
| | | | Summary of Progress | Area & Subheading |
| S1 4- | Rice Utilization Investigations-Southern Region | | | |
| S1 4-13 | A study of the distribution of the constituents of rice in consecutive layers of the kernel and of the evaluation of selected fractions and degrees of milling, as a basis for the development of new and/or improved rice products.** | New Orleans, La. | Yes | 10-A-1 |
| S1 4-14 | Research to improve the potential of rice for new food, feed and industrial products in relation to the physical properties and chemical composition of whole and fractionated kernels.* | New Orleans, La. | Yes | 10-A-1 |
| S2 1- | Cotton Utilization Investigations | | | |
| S2 1-195 (Rev.) | Investigation of radiochemical yields of high-energy radiation activated reactions of cotton to develop improved cotton products. | New Orleans, La. | Yes | 1-B-2 |
| S2 1-197 (C)(Rev.) | Evaluation of stretch-type cotton yarns in knit wear.** | Raleigh, N. C. | Yes | 1-C-8 |
| S2 1-200 (C)(Rev.) | Development of weather-resistant, water-repellent finishes for cotton.** | Denton, Texas | Yes | 1-C-4 |
| S2 1-207 | Development of guides for the maximum utilization of cottons of varying fiber properties.** | New Orleans, La. | Yes | 1-B-5 |
| S2 1-208 (Rev.) | Investigation of the effects of gross and fine structures of the cotton fibers on their physical behaviors. | New Orleans, La. | Yes | 1-A-2 |
| S2 1-209 (Rev.) | Microscopical investigations of absorption phenomena in native,,mercerized, and modified cottons.** | New Orleans, La. | Yes | 1-A-1 |
| S2 1-214 (Rev.) | Separation and identification of the cleavage products of partially etherified cottons, including crosslinked cottons. | New Orleans, La. | Yes | 1-A-4 |
| S2 1-217 (C) | Effect of variation in structure on cotton fiber properties caused by environmental and genetic factors to obtain basic information important in optimum utilization of cotton.** | College Station, Texas | Yes | 1-A-2 |
| S2 1-221 (C) | Investigations to determine the effects of fiber extensibility on fiber breakage in mechanical processing. | Knoxville, Tenn. | Yes | 1-A-3 |
| S2 1-222 (C) | Treatment of cotton fibers by sonic energy to obtain basic information required for the development of improved equipment for processing cotton into textiles.** | Carteret, N. J. | Yes | 1-B-5 |
| S2 1-223 (C) | Effect of the soiling environment on the soiling tendency of a series of cotton finishes.** | Washington, D. C. | Yes | 1-B-4 |
| S2 1-224 (C) | Determination of optimum yarn constructions, knitting structures and prefabrication design for producing stretchable articles of knitted cotton wearing apparel by slack mercerization.** | Raleigh, N. C. | Yes | 1-C-8 |
| S2 1-225 (C) | The relationship of molecular size, nature, shape, conformation, and configuration of organic non-aqueous compounds to their swelling power on cotton cellulose. | Brooklyn, N. Y. | Yes | 1-A-1 |
| S2 1-228 (C) | Investigation of the physics of seam pucker in relation to fabric structure to develop improved sewing thread for wash-wear cotton products.** | Atlanta, Ga. | Yes | 1-C-3 |
| S2 1-229 (C) | Development of a method for counting neps in cotton at various stages of textile processing. | South Pasadena, Calif. | Yes | 1-A-7 |

*Initiated during reporting year.

**Discontinued during reporting year.

| Work & Line Project Number | Work and Line Project Titles | Work Locations During Past Year | Line Proj. Incl. In | |
|----------------------------|--|---------------------------------|---------------------|-------------------|
| | | | Summary of Progress | Area & Subheading |
| S2 1-231 (C) | An investigation of the chemical modification of cotton through treatments with reagents in the vapor phase.** | East Greenwich, R. I. | Yes | 1-B-1 |
| S2 1-237 (Gr) | Investigation of the configurational interactions between fibers and yarns in the region of local deformations in woven cotton cloth.** | Cambridge, Mass. | Yes | 1-A-5 |
| S2 1-238 (Gr) | Correlation of surface microtopography of treated and untreated cotton fibers with resistance to soiling of cotton textiles. | Tucson, Ariz. | Yes | 1-B-4 |
| S2 1-239 (C) | Development of wash-wear cotton fabric with improved moisture absorptivity by use of reactive swelling agents. | Birmingham, Ala. | Yes | 1-B-1 |
| S2 1-240 (Gr) | An exploratory study of the crosslinking of chemically modified cotton to obtain cotton fabrics with an optimum combination of resiliency and thermoplasticity. | Princeton, N.J. | Yes | 1-B-1 |
| S2 1-241 (C) | Investigation of factors influencing comfort in cotton apparel fabrics.** | Washington, D.C. | Yes | 1-A-6 |
| S2 1-242 (C) | To determine optimum processing procedures for cotton differing in tensile and elastic properties and relate these properties to mechanical processing performance, yarn, and fabric properties. | Auburn, Ala. | Yes | 1-C-1 |
| S2 1-243 (C) | Development of finishes for cotton fabrics to render them more rapid drying. | Washington, D.C. | Yes | 1-B-1 |
| S2 1-244 (C) | Development of improved coated cotton fabrics with optimum strength-weight characteristics for outdoor uses. | Dedham, Mass. | Yes | 1-C-4 |
| S2 1-245 (C) | The development of weather resistant cotton textiles with improved physical properties by interfacial and graft polymerization. | Birmingham, Ala. | Yes | 1-C-4 |
| S2 1-247 | An investigation of the chemical modification of cotton fabrics using reagents in the form of fogs or aerosols.** | New Orleans, La. | Yes | 1-B-1 |
| S2 1-248 (Gr) | Elucidation of the role of fiber morphology on frictional behavior important in mechanical processing of cotton fibers and in the behaviors of cotton products. | Atlanta, Ga. | Yes | 1-A-5 |
| S2 1-249 | Investigations of cotton fiber-property changes, during mechanical and chemical processes, which are responsible for altered sorption of alkali solution.** | New Orleans, La. | Yes | 1-A-1 |
| S2 1-252 | Development and evaluation of a new machine for opening and blending bales of cotton in any desired proportion in textile mills. | New Orleans, La. | Yes | 1-C-2 |
| S2 1-254 | Development of optimal cotton fabric structures for men's trousers and dress suits. | New Orleans, La. | Yes | 1-C-3 |
| S2 1-256 | Development of multipurpose finishes for outdoor cotton fabrics with improved physical properties. | New Orleans, La. | YYes | 1-C-4 |
| S2 1-258 | Exploratory investigation of reversible chemical reactions to obtain information basic to the development of a commercially feasible reversible crosslink.** | New Orleans, La. | Yes | 1-B-1 |
| S2 1-258 (Rev.) | Exploratory investigation of reversible chemical reactions to obtain information basic to the development of a commercially feasible reversible crosslink for cotton apparel uses.* | New Orleans, La. | Yes | 1-B-1 |
| S2 1-259 | The fixation of antimicrobial agents in cotton fabric by use of zirconium compounds, to impart improved weather resistance. | New Orleans, La. | Yes | 1-C-4 |
| S2 1-260 | Investigation of resistance to edge abrasion in wash-wear cotton and methods for improvement.** | New Orleans, La. | Yes | 1-B-1 |

*Initiated during reporting year.

**Discontinued during reporting year.

| Work & Line Project Number | Work and Line Project Titles | Work Locations During Past Year | Line Proj. Incl. in | |
|-------------------------------------|---|------------------------------------|---------------------------|----------------------|
| | | | Summary of Progress | Area & Subheading |
| S2 1-261 | Investigation of spatial and structural effects of reversible and conventional crosslinks in cotton.** * | New Orleans, La. | Yes | 1-B-1 |
| S2 1-261 (Rev.) | | | | |
| S2 1-263 | Microscopical investigations of chemical substitution and crosslink formation in cotton, to provide information basic to research required for increased utilization of cotton.** | New Orleans, La. | Yes | 1-A-2 |
| S2 1-264 | Investigation of the fluorescence spectra of native and modified cottons, to obtain information needed in the development of improved textile products.** | New Orleans, La. | Yes | 1-A-4 |
| S2 1-266 (C) | Development of a research instrument for accurately and automatically determining length, length distribution and diameter of cotton fibers. | South Pasadena, Calif. | Yes | 1-A-7 |
| S2 1-267 | Wash-wear fabrics of increased strength, durability, and luster by crosslinking fabrics woven of premercerized yarns.** | New Orleans, La. | Yes | 1-C-7 |
| S2 1-268 | Development of new basic information concerning the reactions of cellulose by use of nuclear magnetic resonance and other spectroscopic techniques to facilitate research in the chemical modification of cotton.** | New Orleans, La. | Yes | 1-A-4 |
| S2 1-269 | Investigations to improve the production and the performance characteristics of chemically treated cotton batting.** * | New Orleans, La. | Yes | 1-C-8 |
| S2 1-270 | Investigation of the formation of free radicals in fibrous cotton cellulose and the reaction mechanisms of the activated cellulose with selected reagents, to develop new and improved cotton products.** | New Orleans, La. | Yes | 1-B-2 |
| S2 1-271 | Development of improved insect-proof cotton bags for the storage and shipment of food commodities, to maintain and expand markets for cotton. | New Orleans, La. | Yes | 1-C-9 |
| S2 1-272 | The relation of fiber properties to fabric behavior in chemically treated cotton fabrics. | New Orleans, La. | Yes | 1-B-5 |
| S2 1-273 | Development of a method for removing short fibers and improving fiber parallelization at textile carding machines.** | New Orleans, La. | Yes | 1-C-2 |
| S2 1-274 | The effect of high production carding on fiber length distribution and formation of fiber hooks in cotton.** | New Orleans, La. | Yes | 1-C-1 |
| S2 1-275 | Development of more reliable methods of appraising damage done by abrasive action on all-cotton wash-wear fabrics.** | New Orleans, La. | Yes | 1-A-7 |
| S2 1-276 | Investigation of new and improved x-ray diffraction techniques for the study of the crystalline structure of cotton and chemically modified cotton in contact with various interacting liquids.** | New Orleans, La. | Yes | 1-A-4 |
| S2 1-277 | Investigation of the chemical kinetics of cellulose etherifications to expand utilization of cotton. | New Orleans, La. | Yes | 1-B-3 |
| S2 1-278 | Fundamental investigation of basic actions in cotton textile processing by means of high speed photography. | New Orleans, La. | Yes | 1-C-2 |
| S2 1-279 | Development and evaluation of prototype equipment for feeding cotton to textile cards to produce cotton products with improved physical properties. | New Orleans, La. | Yes | 1-C-2 |
| S2 1-280 | Investigation of accessibility to complexing agents of stable cotton cellulose derivatives. | New Orleans, La. | Yes | 1-A-4 |
| S2 1-281 | New and modified carbamate finishes for deferred cure processing to yield chlorine-resistant, light-fast cotton fabrics. | New Orleans, La. | Yes | 1-C-3 |
| S2 1-282 | Investigations of reactions between cotton cellulose and heterocyclic compounds to develop improved textile products. | New Orleans, La. | Yes | 1-B-1 |

*Initiated during reporting year.

**Discontinued during reporting year.

| Work & Line Project Number | Work and Line Project Titles | Work Locations During Past Year | Line Proj. Incl. in | |
|-------------------------------------|--|------------------------------------|---------------------------|----------------------|
| | | | Summary of Progress | Area & Subheading |
| S2 1-283 | Investigation of new ethers and thioethers of cotton cellulose to develop new commercially attractive textile products. | New Orleans, La. | Yes | 1-B-1 |
| S2 1-284 | Investigation of plastic and oriented states of cotton cellulose to develop permanently shaped wash-wear textiles of long wear life. | New Orleans, La. | Yes | 1-A-2 |
| S2 1-285 | An investigation of the tensile recovery behavior of chemically modified cotton yarns and fabrics to facilitate the development of improved cotton products. | New Orleans, La. | Yes | 1-B-5 |
| S2 1-286 | A study of the blending of treated and untreated cotton fibers as a means of improving properties such as resistance to abrasion in wash-wear apparel thus permitting all-cotton to compete successfully with cotton-synthetic blends. | New Orleans, La. | Yes | 1-C-3 |
| S2 1-287 | A spectroscopic investigation of the molecular changes in structure occurring during the chemical modification of cotton cellulose to facilitate the development of new and improved textile products. | New Orleans, La. | Yes | 1-A-4 |
| S2 1-288 | Improvement of the abrasion resistance of durably creased cotton stretch fabrics and cotton fabrics by preferential crosslinking and polymer deposition treatments. | New Orleans, La. | Yes | 1-C-3 |
| S2 1-289 | Stable crosslinking agents suitable for use in delayed-cure processes for cotton | New Orleans, La. | Yes | 1-C-3 |
| S2 1-290 (C) | Investigation of the effect of resin thermoplasticity or thermosetability on the resistance of treated cotton fabrics to abrasion. | Madison, Wisc. | Yes | 1-B-1 |
| S2 1-291 | Development of basic information on the relationship between the spectral properties of selected chemicals and their suitability for improving cotton's properties. | New Orleans, La. | Yes | 1-A-4 |
| S2 1-292 | Treatment of cotton with finishes containing selected lead and other metal compounds to impart specific end use properties. | New Orleans, La. | Yes | 1-B-1 |
| S2 1-293 (C) | Investigation of polymer encapsulation of cotton fibers to provide new and useful textile products.* | Bronx, N. Y. | Yes | 1-B-1 |
| S2 1-294 | Study of mechanisms involved in producing dry and wet crease resistant cottons by esterification with derivatives of monobasic acids. | New Orleans, La. | Yes | 1-B-3 |
| S2 1-295 | To determine the interaction of processing variables with yarn properties and end breakage in spinning at high processing speeds to produce cotton products of required properties at lowest cost. | New Orleans, La. | Yes | 1-C-1 |
| S2 1-296 | Improved abrasion resistance in cotton fabrics from crosslinking of partially swollen cotton. | New Orleans, La. | Yes | 1-C-3 |
| S2 1-297 | Improvement of the chemical and physical properties of cotton by the incorporation of polyfluorinated compounds.* | New Orleans, La. | Yes | 1-C-5 |
| S2 1-298 | Exploratory research on the use of selected compounds to impart flame, wrinkle, stain and abrasion resistance to cotton products.* | New Orleans, La. | Yes | 1-C-6 |
| S2 1-299 | Exploratory investigation of structural parameters of crosslinks in cotton modified by chemical treatment.* | New Orleans, La. | Yes | 1-A-4 |
| S2 1-300 | Fixation of resilient polymer films on cotton textiles to impart wash-wear properties, durable shape, and increased wear life.* | New Orleans, La. | Yes | 1-C-3 |
| S2 1-301 | A study of the influence of restraint, both internal and external, on the capacity of cotton fibers to sorb swelling solutions.* | New Orleans, La. | Yes | 1-A-1 |

*Initiated during reporting year.

| Work & Line Project Number | Work and Line Project Titles | Work Locations During Past Year | Line Proj. Incl. in | |
|----------------------------|--|---------------------------------|---------------------|-------------------|
| | | | Summary of Progress | Area & Subheading |
| S2 1-302 | Microscopical evaluation of cottons modified by treatment with reagents in the vapor phase.* | New Orleans, La. | Yes | 1-A-2 |
| S2 1-303 (C) | Development of mechanical-chemical surface treatments for fabrics to improve abrasion resistance of durably pressed cotton garments.* | Auburn, Ala. | No | |
| S2 1-304 (C) | Development of methods for improving the dimensional stability of abrasion resistant, durable press fabrics made from blends of resin treated and untreated cotton fibers.* | Dedham, Mass. | Yes | 1-C-3 |
| S2 1-305 | Development of flame retardant cottons with improved abrasion resistance and durable press properties.* | New Orleans, La. | Yes | 1-C-6 |
| S2 1-306 | Development of all cotton fabrics suitable for use in abrasion resistant durable press garments through the use of polymers in combination with crosslinking agents.* | New Orleans, La. | Yes | 1-C-3 |
| S2 1-307 | Investigation of the molecular orientation of native and modified cotton fibers and its relationship to the physical properties of modified cotton.* | New Orleans, La. | Yes | 1-A-2 |
| S2 1-308 | The development of improved durable press, all-cotton fabrics through the application of polymers to cotton yarns and through the development of better fabric structures.* | New Orleans, La. | Yes | 1-C-3 |
| S2 1-309 | Investigation of native and modified cottons by wide line nuclear magnetic resonance spectroscopy to obtain information needed in the development of improved textile products.* | New Orleans, La. | Yes | 1-A-4 |
| S2 1-310 | Study of cellulose reorganization techniques for preparing stabilized durable-press cotton fabrics.* | New Orleans, La. | Yes | 1-C-3 |
| S2 1-311 | The improvement of abrasion resistance and wash-wear performance of durably pressed cotton goods by control of polymer deposition and crosslinking of the cotton cellulose.* | New Orleans, La. | Yes | 1-C-3 |
| S2 1-313 | Development of optimum textile processing techniques, blends, and products for increasing the utilization of low and high Micronaire reading cottons.* | New Orleans, La. | No | |
| S2 1-314 | Investigation of the free radical reaction mechanisms of cotton cellulose with selected chemical reagents to develop new and improved cotton products.* | New Orleans, La. | No | |
| S2 1-315 | Development of a method and prototype equipment for producing a continuous and uniform supply of completely individualized cotton fibers.* | New Orleans, La. | No | |
| S2 1-316 | Investigation of short and long term duration abrasion tests and correlation of both types with actual service wear of all cotton durable press garments.* | New Orleans, La. | No | |
| S2 1-318 | Investigation of the changes in microporous structures of cotton fibers during modification processing.* | New Orleans, La. | No | |
| S3 2- | Citrus and Other Fruit Utilization Investigations-Southern Region | | | |
| S3 2-43 | Investigations on conditions for drying as related to the storage stability and quality of "foam-mat" dried citrus products.** | Winter Haven, Fla. | Yes | 6-B-1 |
| S3 2-44 (C) | Composition of flavor components of peaches (with emphasis on existing commercial varieties in the Southeastern United States).** | Experiment and Athens, Ga. | Yes | 11-A-1 |
| S3 2-46 (C) | Development of practical and efficient pilot plant process for the manufacture of enzymatically debittered grapefruit juice and products with improved flavor, product stability, and storage characteristics. | Lake Alfred, Fla. | Yes | 6-B-2 |

*Initiated during reporting year.

**Discontinued during reporting year.

| Work & Line Project Number | Work and Line Project Titles | Work Locations During Past Year | Line Proj. Incl. in | |
|----------------------------|--|---------------------------------|---------------------|-------------------|
| | | | Summary of Progress | Area & Subheading |
| S3 2-47 | Identification of recently isolated flavones and other neutral orange peel constituents and evaluation of their relation to bitterness and harshness in orange products.** | Winter Haven, Florida | Yes | 6-A-3 |
| S3 2-48 | Study of the composition of essential oils in citrus products, particularly orange, to provide a basis for improvement in quality and uniformity of citrus products.** | Winter Haven, Florida | Yes | 6-A-2 |
| S3 2-49 | Processed grapefruit products of greater attractiveness to the consumer, through exploration of means to prevent or minimize the formation of bitter components in the fruit. | Winter Haven, Florida | Yes | 6-A-3 |
| S3 2-50 | A study of off-flavor development in processed citrus juice in relation to the lipid composition of the suspended matter.** * | Winter Haven, Florida | Yes | 6-A-4 |
| S3 2-51 | Influence of seasonal variations in color and flavor of Texas colored grapefruit on the quality of processed products made therefrom. | Weslaco, Texas | Yes | 6-A-3 |
| S3 2-52 | Factors affecting the stability of a freeze-dried avocado salad product with emphasis on the lipid constituents.* | Weslaco, Texas | Yes | 6-B-3 |
| S3 2-53 | Application of freeze drying to citrus and other subtropical fruits to develop new and improved products.* | Winter Haven, Florida | Yes | 6-B-3 |
| S3 2-54 | Evaluation of the flavor contribution of peel flavones to orange juice and identification of unknown peel flavones to develop improved orange juice products.* | Winter Haven, Florida | Yes | 6-A-3 |
| S3 2-55 | Investigation of the composition of essential oil in citrus products, particularly orange, to afford a basis for maintaining and improving the quality and uniformity of citrus products.* | Winter Haven, Florida | Yes | 6-A-2 |
| S3 2-56 (C) | A study of non-enzymic browning in model systems to develop methods of blocking the reactions involved in flavor deterioration of orange juice crystals.* | Columbus, Ohio | No | |
| S3 2-57 | Development of comminuted whole-fruit, powders, pastes, purees, and beverage products from oranges and grapefruit to improve processing efficiency and to diminish pollution problems.* | Weslaco, Texas | No | |
| S3 2-58 (C) | Development of an oxalate or other organic acid index for estimating the amount of peel solids in orange and grapefruit paste, puree, and juice products.* | South Pasadena, Calif. | No | |
| S3 2-59 | Investigations to improve the commercial utility of the foam-mat process for production of citrus crystals.* | Winter Haven, Florida | Yes | 6-B-1 |
| S3 2-60 | Study of the enzyme reactions in fresh and processed citrus to increase the natural flavor of citrus products.* | Winter Haven, Florida | Yes | 6-A-1 |
| S3 5- | Sweetpotatoes, Cucumbers, and Other Vegetable Utilization Investigations-Southern Region | | | |
| S3 5-23 (Rev.) | Application of new basic information on the chemical constituents of celery stalk (petiole) to the development of processed products of improved flavor and convenience.** | Winter Haven, Florida | Yes | 7-C-3 |
| S3 5-24 (Gr) | Elucidation of molecular structure and chemical characteristics of the pectinase inhibitor in sericea forage and other plant sources. | Durham, N. C. | Yes | 7-A-2 |
| S3 5-27 | Adaptation of laboratory pure culture fermentation procedures to a commercially feasible process for the manufacture of pickled vegetable products. | Raleigh, N. C. | Yes | 7-C-1 |

*Initiated during reporting year.

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|----------------------------|--|---------------------------------|---------------------|-------------------|
| | | | Summary of Progress | Area & Subheading |
| S3 5-28 | Investigations of the chemistry and biochemistry of the carotenoid pigments in fruits and vegetables to facilitate the development of improved and more attractive processed products. | Raleigh, N. C. | Yes | 7-A-1 |
| S3 5-29 | Investigation of effects of cucumber substrate, bacterial species and other environmental factors on the flavor and aroma of cucumbers and fermented cucumber products.** | Raleigh, N. C. | Yes | 7-B-1 |
| S3 5-30 | Application of innovations in food technology to the development of improved products from selected southern-grown vegetables. | Weslaco, Texas | Yes | 7-C-1 |
| S3 5-31 | Incorporation of protein concentrates and other food materials and study of carbohydrate transformation during storage and processing to develop new and improved sweetpotato products.* | Raleigh, N. C. | Yes | 7-C-2 |
| S4 1- | Cottonseed, Peanut and Other Oilseed Investigations-Southern Region | | | |
| S4 1-109 (Rev.) | Investigations of the phospholipid, plasmogen and other lipid or lipid-soluble constituents of peanuts and processed peanut products to expand the utilization of food grade peanuts.** | New Orleans, La. | Yes | 5-B-1 |
| S4 1-112 (C) | Investigations of chemical transformations of fat and terpene olefinic compounds by hydroboration and suitable subsequent reactions to produce useful products.** | Lafayette, Ind. | Yes | 2-A-3 |
| S4 1-116 | Isolation, identification, evaluation, and control of fungi and toxic fungal metabolites which may develop during processing of cottonseed and peanuts to improve the acceptance of their processed products.** | New Orleans, La. | Yes | 2-B-1 5-C-1 |
| S4 1-117 (C) | Development of practical processing methods for inactivation of cyclopropene groups in cottonseed meal that decrease its value as a feed for laying hens. | Chicago, Ill. | Yes | 3-C-1 |
| S4 1-118 (C) | Development of peanut products for use in preparation and fortification of processed and convenience foods to extend usefulness of peanuts.** | Auburn, Ala. | Yes | 5-D-2 |
| S4 1-119 (C) | A study of the relation of the carbohydrate, amino acid and protein components of the peanut to the formation of flavor and aroma during roasting, with the objective of expanding the direct utilization of this commodity. | Stillwater, Okla. | Yes | 5-B-1 |
| S4 1-120 (C) | Development of processing methods using peanuts of known history with respect to different growing, harvesting, and curing conditions that will provide processed peanut products of high quality and free of mycotoxins.** | College Station, Texas | Yes | 5-C-1 |
| S4 1-121 (C) | Study of the limiting environmental conditions for the elaboration of mycotoxins in peanuts to develop information needed to assure the processing of highest quality peanuts. | Auburn, Ala. | Yes | 5-C-1 |
| S4 1-124 | Preparation and evaluation of N-disubstituted fatty amides considered potentially useful as plasticizers, nitrile rubber softeners, and antifungal agents, to develop information basic to the increased utilization of cottonseed and other seed oils assigned to SU.** | New Orleans, La. | Yes | 4-B-1 |
| S4 1-125 | Improvement of processes for making cocoa butter-like fats from cottonseed and peanut oils and the development of data and processing techniques for improving the performance of confectionery fats.** | New Orleans, La. | Yes | 2-C-1 |

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|----------------------------|---|---------------------------------|---------------------|-------------------|
| | | | Summary of Progress | Area & Subheading |
| S4 1-126 | Development of low-fat peanuts having acceptable peanut flavor and texture characteristics.** | New Orleans, La. | Yes | 5-D-2 |
| S4 1-127 | Investigation to determine processing conditions for the production of oils and meals of maximum quality from glandless cottonseed.** | New Orleans, La. | Yes | 3-C-1 |
| S4 1-127 (Rev.) | Investigation to determine the processing conditions needed to produce from glandless cottonseed, oils and meals having maximum utility and value.* | New Orleans, La. | No | |
| S4 1-128 | Development of new and improved techniques for preparing useful derivatives of cottonseed and peanut oils by esterification and interesterification.** | New Orleans, La. | Yes | 2-C-1 |
| S4 1-128 (Rev.) | Development of new and improved processes for preparing commercially valuable esters from cottonseed and peanut oil fatty acids.* | New Orleans, La. | Yes | 2-C-1 |
| S4 1-129 | Investigation of methods for correlating and predicting solubilities of homologous and analogous long chain saturated and unsaturated fatty acid derivatives.** | New Orleans, La. | Yes | 2-A-3 |
| S4 1-130 | Investigation of the chemical composition and characteristics of the protein systems of cottonseed to serve as a basis for improvement of nutritive value of cottonseed meal and flour.** | New Orleans, La. | Yes | 3-A-2 |
| S4 1-132 (C) | Study of sterilizing or inactivating treatments in conjunction with artificial drying and curing of peanuts to develop processing conditions needed for producing mycotoxin-free roasted peanut products of optimum quality. | Stillwater, Okla. | Yes | 5-D-3 |
| S4 1-133 | Inactivation or removal of aflatoxins from contaminated cottonseed, peanuts, and their products to permit utilization in foods and feeds. | New Orleans, La. | Yes | 3-B-1 5-C-1 |
| S4 1-134 (Gr) | The development of procedures for synthesizing labeled malvalic acid esters. | Boston, Mass. | Yes | 2-A-3 |
| S4 1-135 | Isolation of the cyclopropenoid fatty acid in cottonseed and cottonseed oil and the investigation of those chemical and physical properties of the cyclopropenoids important to the preparation and use of cottonseed products. | New Orleans, La. | Yes | 2-A-3 |
| S4 1-136 | Isolation and identification of cottonseed constituents that cause mortalities among swine. | New Orleans, La. | Yes | 3-B-1 |
| S4 1-137 (C) | Biological studies of cyclopropenoid derivatives and cottonseed oils treated to remove cyclopropenoids to assure the production of wholesome commercial cottonseed oils. | St. Louis, Mo. | Yes | 2-A-3 |
| S4 1-138 (C) | Isolation and identification of the reaction products of gossypol with simple esters and model triglycerides | Savannah, Ga. | Yes | 2-A-2 |
| S4 1-139 | Development of suitable processes and processing conditions for inactivation of aflatoxin in cottonseed and peanut products using basic nitrogen compounds. | New Orleans, La. | Yes | 3-C-1 5-D-3 |
| S4 1-140 | The effect of chemical structure of N,N-disubstituted fatty amides on their performance as plasticizers, lubricants, pesticides, antimycotic agents and in other industrial applications.* | New Orleans, La. | Yes | 4-B-1 |
| S4 1-141 (C) | Investigation of the digestion and metabolism of gossypol by poultry and swine; to obtain basic information needed to produce cottonseed meals fully suitable for use in rations for these animals.* | College Station, Texas | No | |
| S4 1-142 | Preparation and characterization of cocoa butter-like fats from the stearine obtained in the solvent winterization of cottonseed oil.* | New Orleans, La. | Yes | 2-C-1 |

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|----------------------------|--|---------------------------------|---------------------|-------------------|
| | | | Summary of Progress | Area & Subheading |
| S4 1-143 | Development of new phase-relation and solubility data for unsaturated fatty esters and other fatty acid derivatives to obtain basic information needed to increase utilization of cottonseed oil.* | New Orleans, La. | Yes | 2-A-3 |
| S4 1-144 | Development of new and improved low-fat peanut products and processes for their manufacture.* | New Orleans, La. | No | |
| S5 2- | Naval Stores Investigations-Southern Region | | | |
| S5 2-46 (C) | Synthesis of terpene alcohols and glycols for use in the production of new and useful terpene derived polymers.** | Ithaca, N. Y. | Yes | 8-B-2 |
| S5 2-49 (C) | Investigation of the acid-catalyzed dimerization of alpha-pinene. | Gainesville, Fla. | Yes | 8-B-1 |
| S5 2-52 | The preparation and reactions of epoxides and ozonization products of resin acids and their derivatives, to explore potential new industrial uses.** | Olustee, Fla. | Yes | 8-B-2 |
| S5 2-53 | Development of improved polyester resins from resin acids.** | Olustee, Fla. | Yes | 8-B-2 |
| S5 2-55 | The preparation of polymerizable monomers for vinyl and condensation-type polymers from terpene acids and terpene acid derivatives.** | Olustee, Fla. | No | |
| S5 2-56 | Conversion of rosin, resin acids and pine gum derivatives into polyols for use in polyurethane applications. | Olustee, Fla. | Yes | 8-B-1 |
| S5 2-57 | Production of useful chemicals from terpenes and resin acids by free radical addition of functional groups. | Olustee, Fla. | Yes | 8-B-1 |
| S5 2-58 | High temperature rearrangements of gum rosin to give new materials of potential industrial utility.* | Olustee, Fla. | Yes | 8-B-1 |
| S5 2-59 (C) | Investigations to expand the use of gum rosin and its derivatives in the adhesives industry.* | Columbus, Ohio | Yes | 8-B-2 |
| S5 2-60 | Isolation and characterization of the major unidentified components of pine oleoresin and rosin to aid the increased utilization of gum naval stores.* | Olustee, Fla. | Yes | 8-A-1 |
| S5 2-61 | The investigation of the chemistry of photoperoxides of pine gum to develop useful intermediates for the chemical industry.* | Olustee, Fla. | Yes | 8-B-2 |
| S5 5- | New and Replacement Crops Utilization Investigations-Southern Region | | | |
| S5 5-51 | Investigations of the chemical characteristics of new sweet sorghum canes which determine their suitability for sugar recovery.** | Weslaco, Texas | Yes | 9-A-1 |
| S5 5-55 | Investigations of methods for purifying sweet sorghum juices which will permit practical recovery of their sugar content.* | Weslaco, Texas | Yes | 9-A-1 |
| SU-0-0-3 (AID) | A study of the preparation of cottonseed and peanut flours and their derived products for human consumption in developing countries. | New Orleans, La. | Yes | 2-C-2 5-D-1 |
| SU-0-0-4 (SG) | Development of cottonseed oil emulsions suitable for extended use in intravenous nutrition. | New Orleans, La. | Yes | 2-C-1 |
| SU P 1 | Seed Protein Pioneering Research Laboratory.*** | New Orleans, La. | Yes | 5-A-1 |
| SU P 2 | Plant Fibers Pioneering Research Laboratory.*** | New Orleans, La. | Yes | 1-A-4 |

*Initiated during reporting year.

**Discontinued during reporting year.

***There are no line projects under this work project.

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|----------------------------|---|---------------------------------|---------------------|-------------------|
| | | | Summary of Progress | Area & Subheading |
| UR-A7-(40)-3 | A study of the relationship of substituent fatty acid groups on the physical properties of diacid triglycerides of palmitic and stearic acids as a means of increasing the utilization of cottonseed oil for food and industrial purposes.** | Bombay, India | Yes | 2-A-3 |
| UR-A7-(20)-4 | Investigation of the photochemical degradation of cotton to derive information which would enhance the utilization of cotton.** | Bombay, India | No | |
| UR-A7-(20)-19 | A study of the relation between fine structure and mechanical properties of cotton fibers by swelling and stretching treatments, as a means of improving the properties, and thereby increasing the utilization of cotton. | Ahmedabad, India | Yes | 1-A-4 |
| UR-A7-(40)-24 | An investigation of chemical transformations of saturated fatty derivatives to <u>alpha</u> , <u>beta</u> -olefinic or <u>tert</u> -olefinic compounds, followed by hydroboration and suitable subsequent reactions, to produce useful products from cottonseed and new crop oils.* | Bangalore, India | No | |
| UR-A7-(40)-26 | Studies of the addition of carbenes to unsaturated fatty materials derived from cottonseed oil, to provide possible new outlets for the utilization of cottonseed oil. | Bangalore, India | Yes | 4-A-1 |
| UR-A7-(40)-28 | Investigation of the synthesis and properties of new-type glycol monoalkyl ethers for the control of water evaporation, to extend the industrial utilization of cottonseed oil. | Poona, India | Yes | 4-A-1 |
| UR-A7-(20)-30 | Investigation of new solvents for molecular weight determination of cellulose, to obtain basic information needed to improve cotton products and thereby enhance the utilization of cotton.** | Bombay, India | No | |
| UR-A7-(20)-32 | Investigation of the microbial decomposition of cellulose with special reference to the effect of Indian bacterial organisms on cotton and cotton fabrics to provide basic information for the improvement of cotton products. | Bombay, India | Yes | 1-A-3 |
| UR-A7-(20)-33 | Investigations of the preparation of radioresistant and radiosensitive celluloses to obtain basic information needed for useful applications of high energy radiation in cotton textile processing, thereby enhancing the utilization of cotton. | Bombay, India | No | |
| UR-A7-(20)-46 | A study of the physical chemistry and thermodynamics of solution and vapor phase adsorption on and in the cotton fiber to obtain basic information needed to improve cotton processing and utilization. | Ahmedabad, India | Yes | 1-A-1 |
| UR-A7-(20)-51 | Investigation of means to minimize fiber hooked ends in cotton card and drawing slivers to develop processing organizations of optimum efficiency, and thus to promote increased utilization of cotton. | Ahmedabad, India | Yes | 1-B-5 |
| UR-A7-(20)-59 | An investigation of moisture sorption and desorption by crosslinked cotton celluloses over the entire humidity range, in relation to the state of swelling under which the cellulose is crosslinked and to other properties of the crosslinked celluloses, to obtain basic information of value in increasing the textile uses of cotton. | Delhi, India | Yes | 1-A-1 |
| UR-A7-(40)-79 | An investigation of the fractionation of hexane-acetone-water mixtures, to obtain fundamental information needed in the design of solvent recovery systems for use in an improved mixed solvent extraction process for extraction of oil from cottonseed.* | Madras, India | No | |

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|----------------------------|---|---------------------------------|---------------------|-------------------|
| | | | Summary of Progress | Area & Subheading |
| UR-A7-(20)-84 | An investigation of heat and mass transfer rates and other basic engineering concepts as related to the drying and curing of resin-treated cotton textiles by countercurrent solid-gas contact systems, to obtain fundamental information necessary to devise more efficient textile processing techniques, thereby increasing the utilization of cotton in textile applications. | Delhi, India | Yes | 1-B-5 |
| UR-A7-(20)-85 | An investigation to determine the factors that affect the drafting capacity, optimum conditions, spinning efficiency, and yarn quality of the direct sliver spinning system to provide information needed to improve cotton processing and increase the utilization of cotton products. | Ahmedabad, India | Yes | 1-B-5 |
| UR-A7-(20)-87 | Investigation of the correlation between several important physical properties of woven cotton apparel fabrics and their performance in actual service tests to obtain information needed for the improvement of cotton textiles. | Delhi, India | Yes | 1-B-5 |
| UR-A7-(20)-109 | An investigation of the effects of atmospheric conditions during the spinning of cotton yarns on yarn properties and spinning efficiency, to obtain basic information needed for improving the utilization of cotton.* | Coimbatore, India | No | |
| UR-A7-(20)-120 | A study of factors affecting curling and bursting of preponderantly warp- and filling-faced cotton fabric structure during processing of cotton into end-use products. | Bombay, India | No | |
| UR-A7-(40)-124 | A study of the synthesis and properties of pure saturated diacid and triacid triglycerides for use as model compounds in obtaining basic information needed to improve the utilization of cottonseed oil. | Bombay, India | Yes | 2-A-3 |
| UR-A7-(20)-132 | Development of a stochastic model for determining the efficiency of drafting independent of the size of fibrous strands, to obtain basic information needed to improve the processing of cotton into useful textile materials.* | Ahmedabad, India | No | |
| UR-A10-(40)-34 | Investigation of π -complexed organometallic compounds derived from polyunsaturated fatty acids, to obtain fundamental information needed in expanding the utilization of cottonseed oil. | Haifa, Israel | Yes | 4-A-1 |
| UR-A10-(20)-50 | A fundamental study of the oxidation of cotton and crosslinked cotton by hypochlorite, hypobromite, and other oxidizing agents, to obtain information needed on the kinetics of the oxidation and the changes in physical and chemical properties, in order to improve the characteristics of cotton for various end uses. | Jerusalem, Israel | Yes | 1-B-3 |
| UR-A10-(40)-52 | Development of methods for the improved preparation of protein hydrolysates for the determination of amino acids, to provide a more accurate means for assessment of protein quality and nutritive value of oilseed proteins, thus contributing to their increased utilization. | Jerusalem, Israel | Yes | 2-A-1 |
| UR-A10-(40)-53 | A study of the preparation of new chemical derivatives from acrylonitrile and fatty acids derived from the oils of cottonseed, tung, parsley seed, <i>Limnanthes douglasii</i> , <i>Cuphea</i> , and other oilseeds of the southern region of the United States to obtain information leading to potential new uses for these oils. | Jerusalem, Israel | Yes | 4-A-1 |

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|----------------------------|---|------------------------------------|---------------------|-------------------|
| | | | Summary of Progress | Area & Subheading |
| UR-A10-(40)-54 | An investigation of metalation reactions employing alkali and alkaline earth metals and their derivatives for the modification of mono- and dienoic fatty acids to provide increased functionality, thereby leading to possible new uses for cottonseed, <u>Limnanthes douglasii</u> , and umbelliferous oils in industrial applications. | Jerusalem, Israel | Yes | 4-A-1 |
| UR-A10-(20)-56 | The synthesis and determination of the properties of new aziridinyl phosphorus compounds having potential for use in the treatment of cotton to afford new products of increased utility. | Jerusalem, Israel | Yes | 1-B-1 |
| UR-A10-(20)-76 | A fundamental investigation of the geometry of wrinkles as they affect the rating of acceptability of ease-of-care treated cotton fabrics, to obtain information needed for the improvement of such cotton fabrics and thus to promote their increased use.* | Jerusalem, Israel | No | |
| UR-A11-(10)-23 | A study of the distribution of the major proteins of rice within subcellular particles, and the distribution of these particles in the cellular structure of the rice kernel, to obtain basic information needed for developing new and improved rice products and methods for producing them. | Kyoto, Japan | Yes | 10-A-1 |
| UR-A11-(40)-24 | Studies of the biochemical mode of action of aflatoxins and their biodegradation, to obtain basic information needed for control of these toxins in cottonseed, peanuts, and other agricultural commodities that may be exposed to contamination by <u>Aspergillus flavus</u> . | Anjo, Aichi, Japan | Yes | 2-B-1 |
| UR-A11-(40)-29 | An investigation of the chemical composition and reactivity of the nucleic acids of cottonseed, to obtain basic information needed for the increased utilization of this commodity. | Kyoto, Japan | Yes | 2-A-1 |
| UR-E4-(20)-1 | A fundamental study of the nature and origin of reversals in cotton fibers and of their relation to mechanical properties of these fibers, to obtain information needed in the development of improved cotton products. | Ghent, Belgium | Yes | 1-A-2 |
| UR-E10-(20)-2 | Development of an apparatus for counting neps in cotton card web as an aid toward increasing the quality of cotton products.** | Reutlingen-Stuttgart, West Germany | No | |
| UR-E15-(40)-33 | Investigations on the physical and physicochemical properties of cottonseed proteins, to obtain basic information needed for the increased utilization of cottonseed. | Rome, Italy | Yes | 2-A-1 |
| UR-E15-(40)-35 | A study of the mechanism of gossypol toxicity counteraction by L-lysine to gain information needed to permit the increased use of cottonseed products in animal feed. | Milan, Italy | Yes | 3-B-1 |
| UR-E15-(40)-44 | Experimental studies to elucidate the role of cottonseed meal in the induction of hepatoma in rainbow trout to obtain fundamental information concerning the suitability of cottonseed meal for use in rations for this species. | Aosta, Italy | Yes | 3-B-1 |
| UR-E19-(20)-12 | An investigation of the fundamental mechanisms and bonding forces that could be used to improve the tensile strength and other physical properties of cotton textiles, as a means of increasing the utilization of cotton. | Delft, Holland | Yes | 1-A-2 |
| UR-E19-(20)-15 | Fundamental investigations to obtain information needed to predict the performance of cotton yarns during weaving, as a means of increasing the utilization of cotton through improvements in its processing.* | Delft, Holland | No | |

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| | | | Summary of Progress | Area & Subheading |
| UR-E19-(20)-20 | A study of the influence of yarn geometry on the response of cotton fibers to stress and deformation after chemical treatments, to obtain information needed for the improvement of cotton products to increase their utility.* | Delft, Holland | No | |
| UR-E21-(20)-27 | An investigation of the mathematical and theoretical aspects of the relationship between the fiber length distribution of cotton specimens before and after sample preparation to obtain basic information needed to improve cotton processing. | Lodz, Poland | Yes | 1-A-7 |
| UR-E25-(40)-19 | An investigation of the rate of reaction of protein with carbohydrates in peanuts, to provide information leading to improved peanut products, thereby increasing the utilization of this commodity. | Granada, Spain | Yes | 5-A-1 |
| UR-E25-(20)-31 | A study of the measurement of the "total hairiness" of cotton yarn and the determination of the mechanical factors contributing toward its formation, to obtain basic information needed to improve the processing of cotton into textiles. | Barcelona, Spain | Yes | 1-A-5 |
| UR-E25-(50)-36 | Development of new or improved methods of synthesizing, isolating, and purifying selected terpene alcohols for use as standards, to obtain basic information on the composition and properties of products made from pine gum as an aid in developing new industrial uses for naval stores products. | Barcelona, Spain | Yes | 8-A-1 |
| UR-E25-(20)-42 | An investigation of the effect of fiber properties on drafting tenacity during spinning of cotton and the interrelationships between fiber properties, drafting tenacity, yarn properties, and end breakage, to obtain basic information related to processing properties in the utilization of cotton. | Barcelona, Spain | Yes | 1-B-5 |
| UR-E26-(20)-1 | Investigation of the mechanism of crease formation and recovery in ease-of-care treated cotton fabrics to supply fundamental knowledge required for the design of improved textiles, thereby increasing the utilization of cotton. | Gothenburg, Sweden | Yes | 1-B-5 |
| UR-E26-(20)-2 | Fundamental investigation of setting reactions for cotton fabrics and garments, to develop information basic to the improvement of cotton products, thereby increasing the utilization of cotton.** | Gothenburg, Sweden | No | |
| UR-E26-(20)-6 | Basic investigation of the behavior of cotton subjected to aerodynamic forces, for the purpose of improving the processing characteristics of cotton textiles. | Gothenburg, Sweden | Yes | 1-B-5 |
| UR-E27-(20)-2 | A study of the chemistry and structural nature of the bonds formed between formaldehyde and cellulose in formaldehyde-treated cottons to provide basic information needed to improve the utility of the cotton fabrics. | Zurich, Switzerland | Yes | 1-A-4 |
| UR-E29-(20)-55 | A fundamental study of the preparation and properties of phosphazene (phosphonitrilic) and phosphoryl chloride derivatives having potential for reaction with cotton cellulose, to obtain information needed in the development of new useful products from cotton, thus increasing its utilization.** | London, England | No | |
| UR-E29-(20)-65 | A study of the effect of caustic soda and other swelling agents on the fine structure of cotton, to obtain basic information needed to improve cotton products and thereby enhance the utilization of cotton. | Manchester, England | Yes | 1-A-4 |

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|-------------------------------------|---|--|---------------------------|----------------------|
| | | | Summary of Progress | Area & Subheading |
| UR-E29- (20)-78 | Investigation of chemical modification of cotton fabrics involving control of lateral molecular order and distribution of crosslinks, to provide basic information needed to improve the performance characteristics of cotton fabrics as a means of increasing their utilization. | Manchester, England | Yes | 1-A-4 |
| UR-E29- (20)-84 | An investigation of the nature and mechanism of the chemical effects of ultraviolet light on cotton cellulose and related compounds under sensitized and unsensitized conditions, to provide information needed for the improvement of cotton textiles for out-of-doors applications through the protection of cotton cellulose from photodegradation by sunlight.* | Cardiff, Wales | Yes | 1-B-5 |
| UR-01- (40)-2 | An investigation of the chemistry and biological effects of cyclopropenoid compounds that occur in cottonseed and cottonseed products, to obtain basic information needed to improve the utilization of these commodities. | Ryde, New South Wales, Australia | Yes | 2-A-3 |

*Initiated during reporting year.

